

# THE WESTERN CANADA SERIES

## LABORATORY EXPERIMENTS IN CHEMISTRY

N. HENRY BLACK

ADAPTED BY R. H. CLARK



AUTHORIZED BY THE  
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FOR BRITISH COLUMBIA

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LABORATORY EXPERIMENTS  
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# LABORATORY EXPERIMENTS IN CHEMISTRY

TO ACCOMPANY

BLACK AND CONANT'S "PRACTICAL CHEMISTRY"

BY

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## PREFACE

LABORATORY work in any science is fundamental and the study of a text book merely extends and organizes the information which the student gets in the laboratory. The experiments which the student does with his own hands should make real and concrete the fundamental principles of the science. It is also essential for him to perform certain experiments which indicate how these facts and principles are utilized at home and in the commercial and industrial life of the community.

The directions for these experiments are at first very full and detailed, but are gradually abbreviated so as to leave more and more to the student's own initiative and imagination. It is hoped that the directions are so clearly stated and illustrated that the teacher will be largely freed from the necessity of explaining the details of manipulation. Frequent questions (in italics) have been inserted in the directions to focus the attention of the student on the important facts to be observed.

One of the aims of all laboratory work is to arouse the student's spirit and enthusiasm for finding out things by experiment. To encourage this spirit numerous questions, problems, and experiments have been suggested as *optional work*. These may well be used to give elasticity and variety to the laboratory work as well as to emphasize the close connection between chemistry in the school laboratory and chemistry in the household, in the factory, or on the farm.

The instructor is expected to select those experiments which meet the needs of his class. Probably *an average class of beginners in chemistry cannot perform successfully more than*

*forty of these experiments.* It is far better to have a small number of experiments carefully done, well written up, and thoroughly understood, than many experiments carelessly rushed through.

In the present laboratory manual there is little material which is either new or original; but special attention has been paid to selecting suitable experiments and to wording the directions. In this work the author has found the following books especially useful and suggestive: *Elementary Studies in Chemistry* by *Joseph Torrey*, *School Chemistry* by *Wilson and Hadley*, *Laboratory Study of Chemistry* by *Smith and Mess*, and *Laboratory Outline of General Chemistry* by *Alexander Smith*; also the *Syllabus in Chemistry for Secondary Schools* published by the Regents of New York State, and the printed laboratory directions used at Harvard College and at the Massachusetts Institute of Technology. The author is also greatly indebted to Professor James Bryant Conant, Dr. Norris F. Hall, and Mr. Bancroft Beatley, all of Harvard University, for their aid in the correction of the proof sheets.

The apparatus required for the essential experiments is simple and the chemicals are inexpensive. Those experiments marked in the table of contents with a star (\*) deal with fundamental facts and should be in any first-year course.

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## GENERAL SUGGESTIONS

**1. Follow Directions.** Before performing an experiment, read the directions through carefully. Make sure that you understand the object of the experiment. Follow the directions closely; remember that every word is significant.

**2. Notebook.** A careful record of each experiment performed should be kept in a substantial notebook. Record the date, the title of the experiment, and the subheadings and paragraph letters (*e.g.* a, b).

Do not duplicate the printed directions. State briefly in your own words *what you did*, *what you observed*, and *what conclusions you drew*. Answer every direct question by a definite statement. Make a simple labeled diagram of the apparatus.

**3. Quantities of materials.** Do not take more of the materials than the amount specified. To take more means a waste of material and of time.

**4. Disposal of waste.** Such things as matches, filter papers, bits of glass, and unconsumed chemicals should be thrown into the waste jars and not into the sink. If much acid or other liquids are emptied into the sinks, flush it out with plenty of water.

**5. Regulate your burner.** The size of the burner flame should be regulated by the stopcock. A small flame is often better than a large one for heating test tubes and beakers.

**6. Neatness is essential.** Make sure your apparatus is clean. After each day's work clean up your apparatus and put your desk in order.



Beaker



Porcelain crucible  
and cover



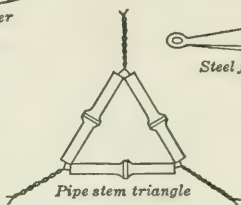
Mortar and pestle



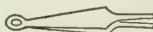
Evaporating dish



Test tube



Pipe stem triangle



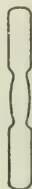
Steel forceps



Ignition tube



Funnel



Spatula



Erlenmeyer flask



Test tube  
brush



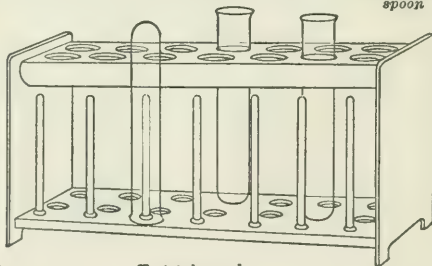
Thistle tube



Deflagrating  
spoon



Florence flask



Test tube rack



Graduated  
cylinder

# LABORATORY EXPERIMENTS IN CHEMISTRY

## HINTS ABOUT LABORATORY MANIPULATION

**Check up your apparatus.** — Make sure that your outfit is complete and in good condition. On the opposite page you will find the names and pictures of some of the less familiar pieces of apparatus. Keep the articles clean and arranged in an orderly fashion.

**Bunsen burner.** — In a laboratory provided with gas the Bunsen burner (Fig. 1) is almost indispensable as a source of heat. When the air holes at the base of the burner are open, air is drawn in and mixes with the gas. It should burn with a clear, blue flame (Fig. 1); the size of the flame can be controlled by adjusting the stopcock. When the holes at the base of the burner tube are closed, a yellow luminous flame results.

In lighting such a burner, never hold the match over it while turning on the gas, but turn on the gas and then apply the match. By the former method it will probably "snap back", that is, burn at the base inside the tube. In this condition the

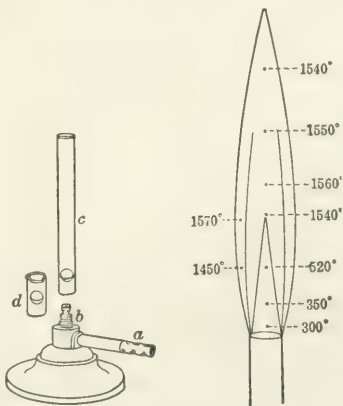


Fig. 1. Bunsen burner and flame.

Figures indicate probable temperatures ( $^{\circ}\text{C}.$ ).

burner gives off a disagreeable gas and becomes extremely hot. Turn off a "snapped" lamp and relight properly.

**Glass tubing.** To cut a piece of small glass tubing, lay it on the table and make a scratch with a triangular file at the point where it is to be cut. Next take the tube in both hands, placing the thumbs on the side of the tube opposite the scratch. Then press the thumbs against the tube and pull the ends of the tube towards you (Fig. 2).

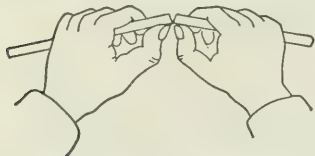


Fig. 2. How to break glass tubing.

To bend glass tubing, rotate it in a flat flame, such as that furnished by the **wing-top** on the

Bunsen burner (Fig. 3), until soft and then take it out of the flame and gradually bend it into the desired shape. In order to make a good bend (Fig. 4), the tube must be heated evenly over a portion which is at least 5 cm. in length. Remember that hot glass placed on a cold table is almost sure to break.



Fig. 3. Heating glass tubing in a flat flame.

Before glass tubes are used for any purpose the sharp edges must be rounded by being heated near the top of a Bunsen flame. This is called "**fire-polishing**" the ends of the tube.



Fig. 4. A good and a bad bend.

**Setting up apparatus.** In putting together apparatus it is often necessary to push a glass tube through a stopper. It is a good plan to lubricate the glass tubing with water, and to push the tube in slowly with a slight twisting motion. It is also very important to leave the joints tight. To test apparatus for gas-tightness, either compress the air in it by blowing or draw some air out by suction. Generally some

part of the apparatus is under water, and so the level of the water can be made to rise or fall in a tube. If there is a leak, this level will change slowly when the opening where the lips were applied is closed. Don't try to plug up holes with matches or wax but *set the apparatus up right*.

**Heating apparatus.** Wipe dry the outside of every glass or porcelain vessel before heating it. In heating a test tube containing a liquid, hold it in an inclined position with some kind of holder and keep it moving in the flame, taking care not to let the flame touch the glass above the liquid. **Don't try to boil a liquid in a flask or beaker with a direct flame.** Use the wire gauze with asbestos center. Turn the flame down until it is just the size of the beaker, and boiling will begin sooner.

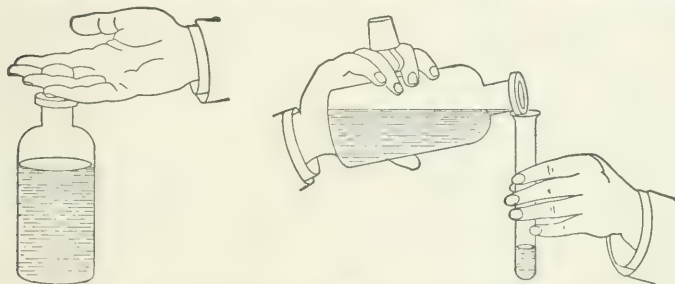


Fig. 5. Proper way to hold a stopper while pouring.

**Porcelain dishes** are used chiefly in evaporating and should be heated over asbestos. A porcelain crucible may be supported on the pipstem triangle and heated directly provided it is warmed up gradually and cooled off slowly.

**Handling materials.** Don't try to *pour solids* out of a bottle. Use some kind of spoon or ladle. Don't weigh materials directly on the scale pans, use a piece of creased paper. Don't use more material than required and don't pour back excess material into the reagent bottle.

**In pouring liquids** from a bottle some care must be taken to prevent the liquid from running down the side of the bottle.

Place the neck of the bottle lightly against the rim of the vessel into which the liquid is being poured. *The stopper must never be laid down on the desk.* Hold it between the fingers as shown in figure 5.

**To filter** a liquid, first fold the filter paper in quarters and then open it up so as to form a cone (Fig. 6). Fit the cone into a funnel of such size that the paper does not quite reach the top and wet it thoroughly with water. The filter is now ready for use.

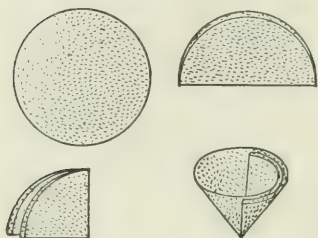


Fig. 6. Folding a filter paper.

**Stirring rods** should be fire-polished. They are very convenient in pouring liquids. The liquid may easily be made to flow down the rod (Fig. 7). A stirring rod should also be used in testing with litmus paper. Put a drop of the liquid to be tested on the paper, don't dip the paper into the liquid.

**Weighing and counting weights.** In weighing out the materials for an experiment, use the platform or trip scales (Fig. 8). If these are properly handled, they are good to one-tenth of a gram (0.1 g.). Remember that the thing to be weighed is put on the *left-hand* pan and the weights on the *right-hand* pan; also remember to use the slider for small weights.

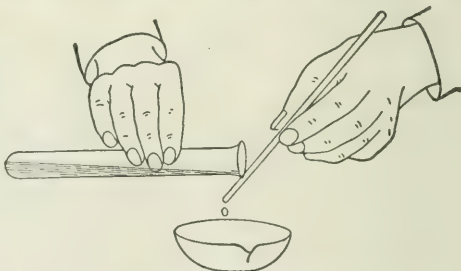


Fig. 7. Pouring a liquid down a glass rod.

For **quantitative experiments** use the horn pan balance (Fig. 9), which is sensitive to one-hundredth of a gram (0.01 g.). A chemical balance (Fig. 10) is usually provided with agate bearings and inclosed in a glass case. Ex-

amine the box of weights and see how they are arranged and marked. Notice that the heavier weights (one gram and above) are made of brass and that the fractional weights are of German silver or aluminum and marked in milligrams. Thus 500 milligrams is 0.5 grams and 10 milligrams is 0.01 grams.

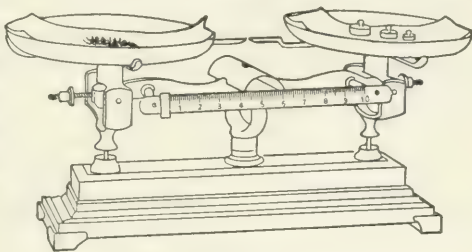


Fig. 8. Platform scales.

In weighing first see that your balance swings evenly, that is, that the pointer swings an equal distance to the right and left. If it does not, adjust it either by means of a set screw or by adding counterbalance. Place the object to be weighed on the left-hand pan and the

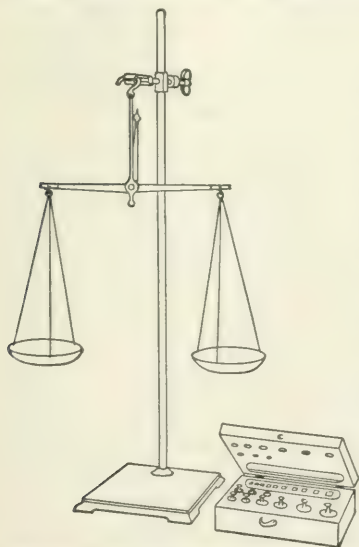


Fig. 9. Horn pan balance and set of weights.

weights on the right. First try a weight which you think is a little heavier than the object; remove it if too heavy and try smaller ones in order. Never handle the weights with your fingers; always use the forceps. Count up the weights on the pan and write down the total at once. *Experience shows that many mistakes are made by miscounting the weights.* Check this result by noting those absent from the box. Beginning with the largest weight, return them to the box and again count them as you replace them.

Do not wait for a balance to come to rest. A good balance

will continue to swing for a long time. Place just enough weights on the pan so that the pointer swings an equal distance to the right and left.

**Measuring volumes.** A graduated glass cylinder (Fig. 11) is commonly used to measure the volume of a liquid. Care

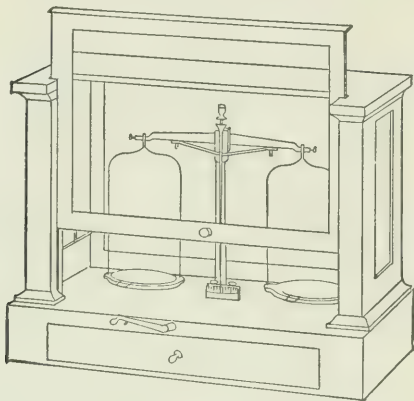


Fig. 10. Chemical balance.



Fig. 11. Graduated glass cylinder.

must be taken to keep the eye on a level with the surface of the liquid. Get familiar with the scale markings and their value.

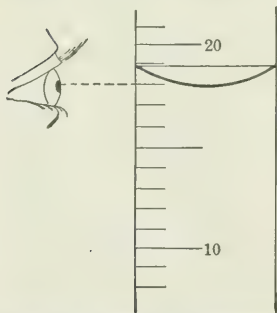


Fig. 12. Reading the meniscus of water.

The eye should follow the scale from the zero end. It will also be noticed that the surface of water is not flat but curved. Such a curved surface is called a **meniscus**. With a concave meniscus, such as occurs on water, the position of the lowest point of the lowest curve against the scale is to be taken as the reading (Fig. 12). In measuring small volumes use a smaller graduated cylinder, a conical graduate, or a graduated test tube.

## HOW TO MAKE NOTEBOOK DRAWINGS

Don't try to make a picture of your apparatus, but make a **sectional drawing**. Imagine the apparatus to be cut by a vertical plane; suppose the sheet of paper to be placed in the position of this plane. Then draw the lines where the paper

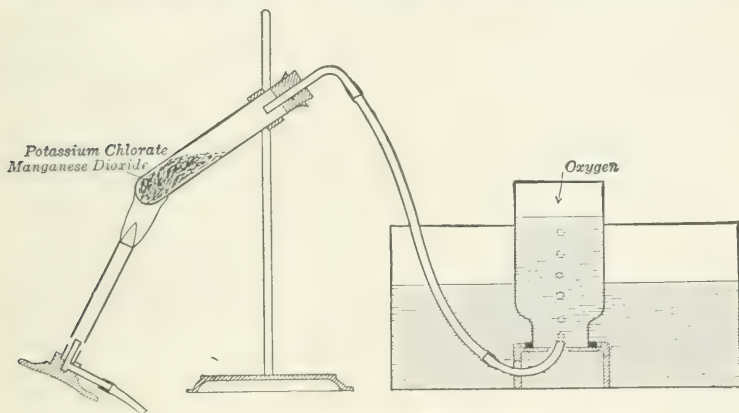


Fig. 13. Sectional drawing of oxygen-generating apparatus.

would cut the intersected apparatus. Figure 13 shows a sectional drawing of the apparatus used to make oxygen. It is also useful for review purposes to label the drawing. Unless you are unusually skillful in free-hand drawing you had better use a ruler.

## EXPERIMENT 1

### CHANGES IN SUBSTANCES HEATED IN AIR

**Apparatus.** — Forceps. Bunsen burner. Iron spoon.

**Materials.** — Copper wire. Magnesium ribbon (6 cm.). Granulated sugar.

a. Examine a piece of bright copper wire and record those characteristic properties which enable one to recognize it as

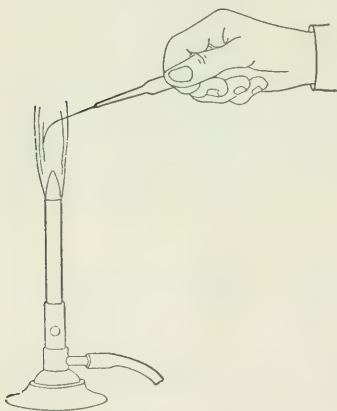


Fig. 14. Heating wire in Bunsen flame..

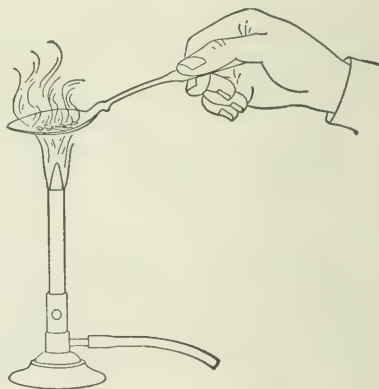


Fig. 15. Heating sugar in an iron spoon.

copper. Hold the wire with forceps in the tip of the flame of a Bunsen burner (Fig. 14) until you are sure that longer heating will produce no further change. Cool and compare the surface material with a piece that has not been heated. *Is the copper changed?*

**NOTE.** Always state the evidence on which you base your conclusions.

**b.** Repeat the experiment (a), using a piece of magnesium ribbon. *Record definite properties of magnesium and of the product, such as: color, appearance (metallic or otherwise), and pliability or brittleness.*

**c.** Heat a bit of sugar (size of a pea) in an iron spoon as shown in figure 15. Note any flame, smoke, or odor produced. *Record the change in appearance and the relative amount of the residue. Identify it by its properties.*

**Optional work.\*** Heat a platinum wire and compare the resulting product with the original wire. *Is it changed?* In the same way examine some tin (foil or granulated), salt, and sulphur, and record all the properties of each which you can easily determine. Heat each substance separately in the air and note what happens as well as the properties of the products.

*Are these substances changed by heat? How do you know? What is the most marked difference between the behavior of sulphur and that of all the other substances? Is the sulphur annihilated?*

## EXPERIMENT 2

### CHANGE IN WEIGHT IN A METAL HEATED IN AIR

**Apparatus.** — Porcelain crucible. Horn pan balance and weights. Ring stand. Pipe-stem triangle. Bunsen burner.

**Materials.** — Copper wire.

**CAUTION.** Never place chemicals of any sort directly upon the scale pans. Never weigh an object while it is hot.

**a.** Weigh a clean, dry porcelain crucible (without cover) to the nearest centigram (0.01 g.). Wind about 5 meters of copper wire around a pencil; place the coil in the crucible and weigh the whole as before. Support the crucible on a pipe-stem triangle (Fig. 16) and heat, at first gently and then with

\* Apparatus and material needed for the optional work are not included in the list at the beginning of the experiment.

the full Bunsen flame, for 15 minutes. The flame should be adjusted so that the crucible is just above the tip of the inner blue cone. For the purposes of this experiment it is not necessary that the action be complete.

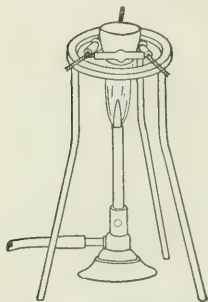


Fig. 16. Heating a crucible on a pipe-stem triangle.

b. To cool the crucible, gradually lower the flame and finally remove it, allowing the crucible to cool on the triangle until it can be held in the hand.

Weigh the crucible again.

*Has there been a loss or a gain in weight? To what is the difference in weight probably due?*

*Record your weights in tabular form as follows:*

Wt. of crucible + copper . . . . .	g.
Wt. of crucible empty . . . . .	g.
Wt. of copper taken . . . . .	g.
Wt. of crucible + copper after heating . . . . .	g.
Wt. of crucible empty . . . . .	g.
Wt. of copper after heating . . . . .	g.
Change in wt. of copper . . . . .	g.

**Optional work.** Repeat the experiment, using instead of copper 2 or 3 g. of powdered iron or granulated tin.

### EXPERIMENT 3

#### SEPARATING A MIXTURE INTO ITS COMPONENTS

**Apparatus.** — Test tubes and corks to fit. Funnel. Filter papers.

Crystallizing dish. Small flask. Evaporating dish. Ring stand.

**Materials.** — Black gunpowder. Carbon disulphide.

a. Examine some black gunpowder. Although it is a mixture of three quite different substances, yet they are so

carefully put together that they are not easily discernible. However, we can separate the various components by using suitable solvents. Carbon disulphide will dissolve the sulphur, water will dissolve the niter or saltpeter, and the charcoal, which is insoluble in both of these solvents, is left behind.

b. Put about 5 g. of gunpowder into a test tube to which a cork has been fitted. Pour upon the gunpowder 10 cc. of carbon disulphide, replace the cork, and shake the test tube for 5 minutes. Allow it to settle, pour off the clear liquid through a dry filter (Fig. 17) into a beaker or crystallizing dish, and set it aside where no flame will get at it.



Fig. 17. Separating a mixture by filtration.

**CAUTION.** Carbon disulphide is very inflammable and must never be handled near a flame.

The carbon disulphide will soon evaporate and leave crystals of sulphur.

c. Spread the black residue left in the test tube on a filter paper to dry. When it has dried, transfer the residue to a small flask. Pour 20 cc. of hot water upon it and shake for 5 minutes. The saltpeter is dissolved, and the solution may be filtered into a clean evaporating dish. By heating the filtrate the water is evaporated, leaving a white residue, the saltpeter or niter.

d. The black residue left on the filter paper is the third component of gunpowder, charcoal or carbon.

*What property of each of the three components of gunpowder was made use of in this separation?*

**Optional work.** Make mixtures of the following substances and then separate the components by means of a suitable solvent:

- (1) Powdered chalk and common salt.
- (2) Powdered zinc and sulphur.
- (3) Sand and sugar.

## EXPERIMENT 4

### DECOMPOSITION OF A COMPOUND

**Apparatus.** — Ignition tube (hard-glass test tube) with 1-hole rubber stopper. Ring stand and clamp. Pneumatic trough. Test tubes. Glass tubing. Bunsen burner. Wing-top.

**Materials.** — Mercuric oxide. Splint.

**a.** Place about 2 or 3 g. of the red powder (mercuric oxide) in the bottom of a clean ignition tube (hard-glass test tube).

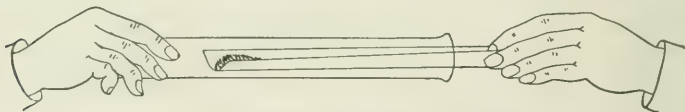


Fig. 18. Sliding a powder into a test tube.

This can be done by putting the powder near the end of a narrow strip of folded paper and sliding it carefully into the tube as shown in figure 18. By inclining the tube and gently tapping the paper, the powder will be deposited in the bottom of the tube, leaving the sides of the tube perfectly clean.

Now fit the ignition tube with a one-hole rubber stopper carrying a delivery tube which is arranged as shown in figure 19. Fill a test tube with water and invert it over the end of the delivery tube in a dish of water (pneumatic trough).

b. Warm the ignition tube very gradually by holding the burner in the hand and waving the flame along the tube. Very

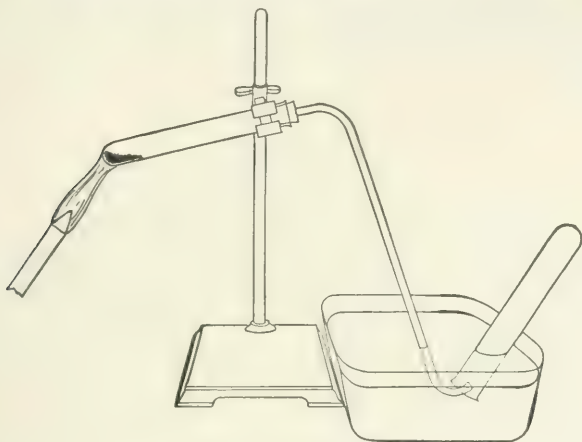


Fig. 19. Decomposing mercuric oxide.

soon bubbles of gas will be seen coming from the end of the delivery tube.

*What is the cause of this bubbling?*

When half a test tube of gas has been collected, remove the test tube, invert it, and test the gas by thrusting into it a glowing (not flaming) splint. *Is the gas air?*

**CAUTION.** Never allow the ignition tube to cool while it is connected with a delivery tube which has its free end under water. *Explain what would happen.*

c. Continue heating the ignition tube until the powder is all decomposed. Collect a second test tube of gas and test with a glowing splint. This gas is oxygen. *What is the difference in the behavior of the glowing splint in the two test tubes?*

d. Scrape off with a splint some of the substance left on the side of the ignition tube. *What is it?*

*Into what two substances has the red powder been decomposed?*

**Optional work.** Place about 1 cc. of lead dioxide in an ignition tube and fasten the tube upright in the clamp on the ring stand. Heat at first gently and then strongly with the Bunsen flame. Test for oxygen by introducing a glowing splint. After removing the flame, examine the residue in the tube. This new substance is **litharge**.

## EXPERIMENT 5

### OXYGEN — PREPARATION AND PROPERTIES

**Apparatus.** — Test tube and 1-hole stopper. Ring stand with clamp. Bunsen burner. 4 wide-mouth bottles. 4 glass squares. Pneumatic trough. Deflagrating spoon. Glass tubing.

**Materials.** — Potassium chlorate. Manganese dioxide. Splint. Splinter of charcoal. Copper wire (15 cm.). Sulphur. Red phosphorus. Asbestos paper.

**I. Preparation.** Mix on paper about 10 g. of potassium chlorate and about one-third the bulk of manganese dioxide. Slide the mixture into a test tube, clamp it horizontally, and tap it so as to distribute the mixture along the side of the lower half of the tube, leaving space above the mixture to the end of the tube. Insert the rubber stopper carrying the delivery tube as shown in figure 20. Fill the pneumatic trough with enough water to cover the support for the bottles. Fill 4 wide-mouth bottles with water, cover them with glass plates, and invert them in the trough.

Use a small Bunsen flame to heat the tube, keep the flame moving, and remove the flame when the gas comes too fast. Heat slowly. *Will the gas which passes over at first be pure oxygen?* Now fill the bottles with oxygen one by one, covering each when full with a glass plate, and setting it mouth upwards on the table. After the fourth bottle is filled, remove the delivery tube from the water and allow the test tube to cool. The residue in the test tube may be easily removed with warm water.

**II. Properties.** *a.* Use one bottle of the gas to determine such properties as color, odor, and solubility, and then thrust in a glowing splint. (Sometimes a slight cloud is present when oxygen is made from potassium chlorate, due to the presence of

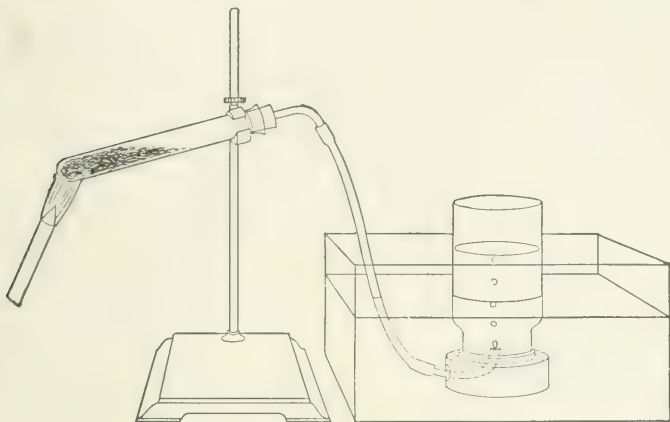


Fig. 20. Decomposing potassium chlorate.

an impurity.) The solubility can be inferred from the method of collecting the gas.

*b. Charcoal.* Wind one end of a piece of wire around a thin splinter of charcoal. Heat the charcoal until it glows brightly and immediately lower it into a bottle of oxygen. *Compare the combustion of charcoal in oxygen with that in air. Of what does the gas produced probably consist?*

*c. Sulphur.* Line a combustion or **deflagrating spoon** with asbestos paper and put in it about 0.5 cc. of sulphur. Ignite the sulphur in the Bunsen flame. Observe the flame for a moment and then lower the spoon into a bottle of oxygen, keeping the mouth of the bottle closed as far as possible with the glass plate. *Compare the flame of sulphur burning in oxygen with that in air. Is it finally extinguished? If so, why? Very cautiously smell the contents left in the bottle. What is it?*

**d. Phosphorus.** Clean and reline the spoon with asbestos. Place in it a bit of red phosphorus (size of small pea), ignite and lower it into another bottle of oxygen. *Compare the burning as before. Is the product a gas, or does it consist of fine solid particles?*

**CAUTION.** Before putting the spoon away, clean it by heating it to redness for two minutes to burn up all the phosphorus.

*Record the properties of oxygen which you have observed in this experiment and its most striking characteristic.*

**Optional work.** Prepare oxygen from sodium peroxide (a white solid containing 41 per cent of oxygen). This can be

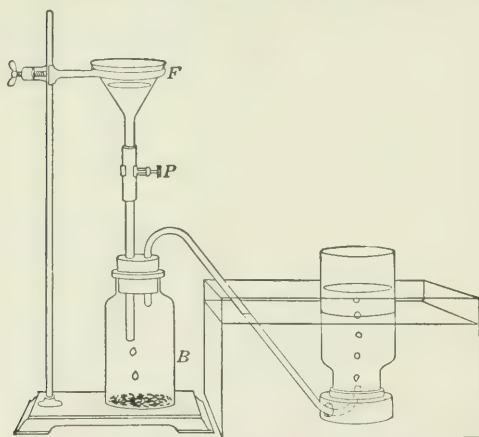


Fig. 21. Making oxygen from sodium peroxide and water.

conveniently done by merely dropping water on the peroxide in the apparatus shown in figure 21. Put about 5 g. of sodium peroxide in the bottom of the bottle *B* and partly fill the funnel *F* with warm water. Open the pinchcock *P* a little from time to time so that the water will fall upon the peroxide. Always keep a little water in the funnel. Collect the oxygen as before.

## EXPERIMENT 6

## PERCENTAGE OF OXYGEN IN POTASSIUM CHLORATE

**Apparatus.** — Porcelain crucible and cover. Ring stand. Horn pan balance. Set of weights. Pipe-stem triangle. Iron forceps. Bunsen burner.

**Materials.** — Dry potassium chlorate.

Clean and dry a porcelain crucible and cover. Weigh the crucible empty to a centigram (0.01 g.). Put about 2 grams of *dry* powdered potassium chlorate in the crucible and weigh again as before.

Place the covered crucible on the pipe-stem triangle and support it on the ring stand at some convenient height (Fig. 22). Begin heating with a small Bunsen flame (about 5 cm. high) very gently. Lift the cover occasionally with the forceps and adjust the flame so that the melted potassium chlorate gives off its oxygen slowly. In case some should spatter up on the cover, withdraw the flame and remove the cover to a clean piece of smooth paper. Loosen the bits of solid matter with a pin and very carefully return them to the crucible. Continue heating until the bubbling slackens, then heat strongly for 10 minutes. Now lower the flame to permit the crucible to cool gradually and finally remove it. When cold, weigh to the nearest centigram (0.01 g.).

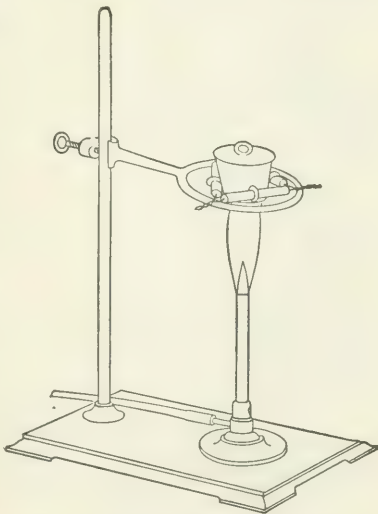


Fig. 22. Heating potassium chlorate in a crucible.

Again heat very hot for about 5 minutes, cool, and weigh as before. If the weight has not changed during the second heating, we may consider that the oxygen has been completely expelled. If the weight is less, heat once again for 5 minutes and continue in this way until two successive weights are the same. This process is called **heating to constant weight**.

The loss of weight is equal to that of the oxygen. The residue left in the crucible is called **potassium chloride**.

The percentage of oxygen in potassium chlorate may be calculated thus:

$$\text{Wt. of potassium chlorate} : \text{wt. of oxygen} :: 100 : x$$

Make all computations in your notebook. Arrange your work in tabular form:

Wt. of crucible + potassium chlorate	. . . . .	g.
Wt. of crucible empty	. . . . .	g.
Wt. of potassium chlorate	. . . . .	g.
Wt. after first heating	. . . . .	g.
Wt. after last heating	. . . . .	g.
Wt. of oxygen	. . . . .	g.

*Find the average of the percentages found by all in the class who have done this experiment. What law of chemistry is thus illustrated?*

**Optional work.** The same method may be used to obtain the percentage of oxygen in sodium chlorate.

# EXPERIMENT 7

## DECOMPOSITION OF WATER BY SODIUM

**Apparatus.** — Knife. Evaporating dish. Forceps. Ring stand and clamp. Pneumatic trough. Large test tube. Glass plate. Stirring rod.

**Materials.** — Sodium. Litmus paper strips, red and blue. Lead foil.

**CAUTION.** The reaction of sodium with water is violent. Don't handle sodium with wet hands. Follow the directions.

a. What is the appearance of a freshly cut surface of sodium? *Is sodium hard or soft? heavy or light?*

b. Fill an evaporating dish one-fourth full of water, and drop into it with the forceps a bit of sodium (size of the head of a match). It is well to protect the eyes from spattering, with a plate of glass (Fig. 23). *Result?*

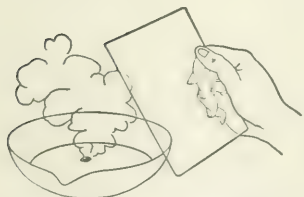


Fig. 23. Violent action of sodium on water.

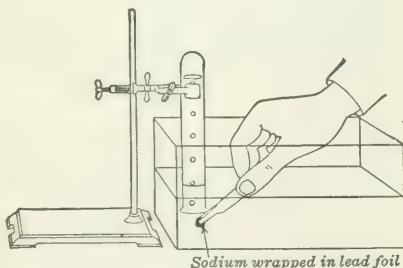


Fig. 24. Collecting the gas evolved from sodium and water.

Throw in another similar piece of sodium. Apply a lighted match, held at arm's length, to the sodium while it is reacting on the water. *Result?*

c. After the action has ceased, wet your fingers with the solution and rub them together. *Result?* Test with a stirring rod the action of a drop of the solution on red and blue litmus paper. *Result?*

d. Wrap a clean, freshly cut piece of sodium (about 3 mm. in diameter) in dry lead foil (tea lead). Punch several holes in the lead with a knife blade. Holding the lead in forceps, quickly slip it under the mouth of an inverted large test tube filled with water (Fig. 24). If necessary, use another piece of sodium wrapped in lead to complete the filling of the tube. If the sodium escapes from the lead covering, stand aside.

When the tube is filled with gas, carry it mouth downward to a flame and ignite the gas. *Result?* This gas is hydrogen, and the yellow flame is due to sodium. *If sodium is an element, where does the gas come from?*

Draw a diagram of the apparatus used in d.

**Optional work.** Calcium is also a metal which liberates hydrogen from water. The reaction is not so violent as that of sodium with water. Hence it is perfectly safe and furnishes a better experiment than sodium. Calcium does not have to be wrapped in lead foil and can be handled much as other common metals.

**NOTE.** Metallic calcium may be cut up into small pieces by fixing it in a vise and chipping it off with a chisel.

## EXPERIMENT 8

### HYDROGEN — PREPARATION AND PROPERTIES

**Apparatus.** — Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. Glass tubing. 3 wide-mouth bottles and glass squares. Bunsen burner. Test tube.

**Materials.** — Zinc. Dilute sulphuric acid. Copper sulphate solution. Wax tapers.

**I. Preparation.** A convenient method of preparing hydrogen is furnished by the action of zinc on dilute sulphuric acid. Place about 20 g. of zinc in a bottle provided with a two-hole rubber stopper carrying a thistle tube and a delivery tube. The apparatus is arranged for collecting the gas over water (Fig. 25).

Insert the stopper tightly with a twisting motion and pour through the thistle tube enough dilute sulphuric acid to cover the zinc. **Be sure that the thistle tube dips into the acid.** If the gas does not come off freely, add through the thistle tube a few drops of copper sulphate solution.

The gas which passes over first is evidently a mixture of hydrogen and air. A confined mixture of hydrogen and air, if ignited, explodes with great violence. To determine when the hydrogen is free from air,

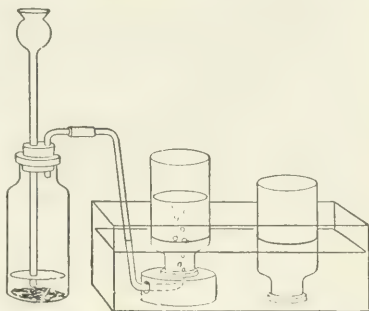


Fig. 25. Generating hydrogen by the action of zinc on dilute sulphuric acid.

collect a test tube full of the gas and carry it mouth downward to a small flame and ignite the gas. Continue to collect and test the gas in this way until a portion does not give a sharp explosion but burns quietly. Then collect 3 bottles of hydrogen, cover each with a glass plate, and set mouth downward on the desk.

**II. Properties.** *a.* Allow one bottle of hydrogen to stand uncovered (mouth up) for a minute. Then bring a lighted taper to the mouth of the bottle. *What does this show about the density of hydrogen as compared with air?*

*b.* Light a short taper and lift a second bottle of hydrogen (mouth downward). Insert the burning taper almost to the bottom of the bottle and then withdraw it slowly. *What happens to the flame of the taper? What occurs at the mouth of the bottle? Explain.*

*c.* Place a bottle of hydrogen, mouth downward, over a bottle of air, mouth upward. Allow the bottles to remain mouth to mouth in this position for 3 minutes. Carry first the lower bottle and then the upper bottle quickly to a flame. *What fact about the diffusion of gases does this experiment illustrate?*

*d. Summarize the properties of hydrogen and compare with those of oxygen.*

**Optional work.** Filter the liquid remaining in the flask (the black specks in the liquid of the generator are impurities, mainly carbon, which were in the zinc), and evaporate to about one-half its volume in a porcelain dish over asbestos. Cool and allow the liquid to stand until crystals form. They are called **zinc sulphate** and contain zinc, sulphur and oxygen. *If sulphuric acid contains hydrogen, sulphur, and oxygen, how would you describe the chemical change that takes place in the gas-generating bottle?*

## EXPERIMENT 9

### REDUCTION OF AN OXIDE WITH HYDROGEN

**Apparatus.** — Wide-mouth bottle with 2-hole stopper. Thistle tube. Drying tube with stopper. Ring stand and clamp. Bunsen burner. Test tube. Glass tubing.

**Materials.** — Copper oxide (wire form). Zinc. Dilute sulphuric acid. Absorbent cotton. Fused calcium chloride.

**a.** We have already seen that bright metallic copper turns to black copper oxide when heated in the air (Exp. 1). In this experiment we shall reverse the process of oxidation and change copper oxide back to metallic copper; this is called **reduction**.

For this purpose we shall use dry hydrogen (free from water vapor) which can be obtained by passing the gas from the generator through a tube filled with granulated calcium chloride. This drying material is held in place by loose plugs of cotton placed at each end of the tube.

**b.** Place about 0.5 cc. of black copper oxide in the bottom of a dry test tube and place the latter in a slightly inclined position with mouth downward (Fig. 26). Insert a straight piece of glass tubing into the stopper of the drying tube and introduce this tubing into the test tube until the end touches the copper oxide.

c. Now generate hydrogen as in Experiment 8, and wait 3 minutes to allow all the air to be replaced by hydrogen.

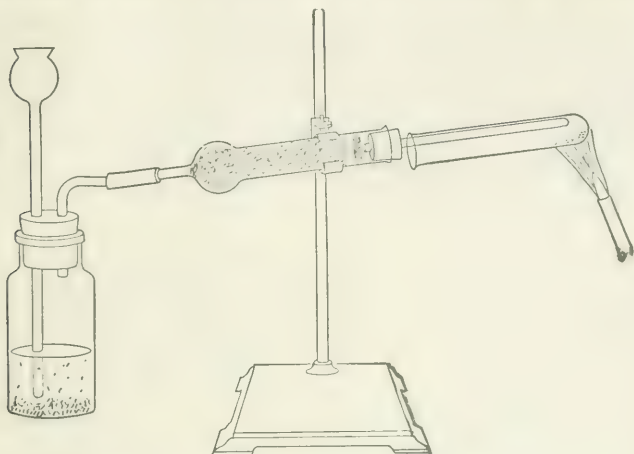


Fig. 26. Reducing copper oxide with dry hydrogen.

**CAUTION.** Have no flame near the apparatus until all the air is driven out.

Cautiously heat the copper oxide to redness, being careful to keep the flame away from the mouth of the tube.

*Note any change in the copper oxide. What collects in the cool part of the tube? What substance is reduced and what substance is oxidized in this experiment?*

*Why is it necessary to dry the hydrogen?*

**Optional work.** Try this experiment using ferric oxide instead of copper oxide.

## EXPERIMENT 10

## WEIGHT OF A LITER OF OXYGEN

**Apparatus.** — Iron crucible. Ring stand and clamp. Pneumatic trough. 2-liter bottle (acid bottle). Test tube with 1-hole stopper. Horn pan balance and weights. Bunsen burner. Glass plate. Graduated cylinder. Glass tubing.

**Materials.** — Manganese dioxide (powdered). Well dried potassium chlorate. Glass wool.

a. In this experiment we first determine the weight of a definite volume of oxygen and then compute the weight of one liter measured under standard conditions. To determine the weight of the oxygen, we weigh the generating apparatus both before and after a definite volume of oxygen has been produced. To measure the volume of the oxygen, we measure the volume of water which it displaces and then, knowing the temperature and barometric pressure, we can compute according to the Gas Laws [§ 47] \* the volume under standard conditions (0° C. and 760 mm.).

b. It is important in this experiment to make sure that the loss of weight in the generating apparatus is due to the oxygen evolved and not in part to moisture driven off from the materials used. Therefore it is well to heat about 6 g. of pulverized manganese dioxide in an iron crucible (uncovered) for 5 minutes, stirring it occasionally.

In the meantime, fill an acid bottle (about 2 liters capacity) with water and invert it in the pneumatic trough. Insert a delivery tube in a one-hole rubber stopper which fits a dry test tube.

When the manganese dioxide has cooled, mix it on a sheet of paper with 7 g. of dry pulverized potassium chlorate. Slide the mixture into the test tube and place a plug of glass wool or

\* Bracketed numbers refer to sections in *Black and Conant's Practical Chemistry*.

well ignited asbestos fiber in the mouth of the tube to catch small particles carried along by the gas. Weigh the test tube with its contents to the nearest centigram (0.01 g.) and record the result according to the following table. Replace the stopper in the test tube and test for air-tightness as follows: place the end of the delivery tube in the mouth and suck out some of the air. If the tip of the tongue seems to adhere to the tube, the apparatus is tight. Arrange the apparatus as shown in figure 27.

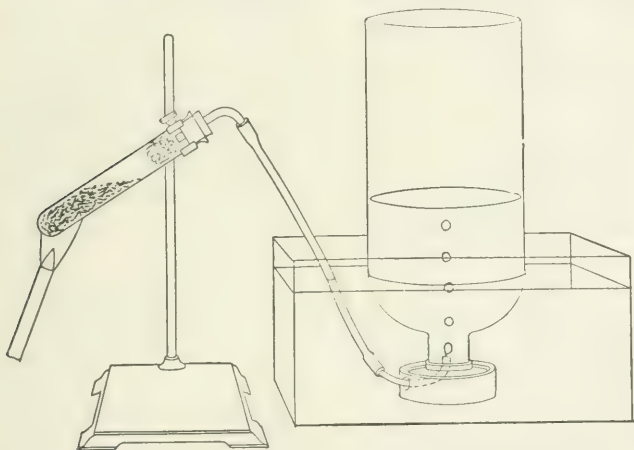


Fig. 27. Apparatus for finding the weight of a liter of oxygen.

**c.** Heat the contents of the test tube with a small flame, beginning at the top and gradually working downward. Collect all the gas evolved in the bottle. **Don't rush! Regulate the heating so that you could count the bubbles of oxygen.**

When the bottle is nearly full, or when the oxygen has stopped coming over, remove the delivery tube from the water and allow the test tube to cool.

**d.** In the meantime, to measure the volume of the oxygen, first equalize the level of the water inside and outside the bottle. To do this, it may be necessary to lower the bottle and incline

it. Then, while the bottle is in this position, close it with a stopper or glass plate and set it upright on the table.

To measure the volume of gas collected in the bottle, measure the amount of water needed to fill the bottle, pouring the water into the bottle from a graduated cylinder. Record the total volume of water used, the temperature of the water in the pneumatic trough, and the barometric pressure.

When the test tube has cooled so that it feels barely warm, weigh the test tube and its contents as before and record the weight. Compute the loss of weight, which is the weight of oxygen evolved.

Correct the barometric pressure for aqueous tension [§ 48] and then reduce the observed volume of oxygen from the observed temperature and pressure to 0° C. and 760 mm.

From this reduced volume and the weight, compute the weight of 1 liter of oxygen (density).

TABLE

Wt. of test tube + contents before heating . . . . .	g.
Wt. of test tube + contents after heating . . . . .	g.
Wt. of oxygen evolved . . . . .	g.
Temperature . . . . .	°C.
Volume of oxygen under conditions of exp. . . . .	cc.
Barometric pressure . . . . .	mm.
Pressure due to water vapor . . . . .	mm.
Corrected barometric pressure . . . . .	mm.
Volume of oxygen under stand. conditions . . . . .	cc.
Density of oxygen (wt. of 1 liter) . . . . .	g.

## EXPERIMENT 11

## DISTILLATION OF WATER

**Apparatus.** — Flask with 1-hole stopper. Ring stand. Wire gauze with asbestos center. Bunsen burner. Wide-mouth bottle. Test tubes. Glass tubing.

**Materials.** — Common salt (sodium chloride). Potassium permanganate. Ammonia water (concentrated). Litmus paper (red). Marble chips or glass beads.

a. Dissolve a spoonful of common salt in about 50 cc. of water. Put the solution in a flask which is arranged as shown in figure 28. A few chips of marble in the flask will prevent "bumping." The steam from the boiling water in the flask is condensed by conducting it into the test tube which is kept cold by the water in the bottle. Boil gently until 5 cc. has distilled over. Taste the distilled water. *Is it free from salt? Explain.*

Repeat using 50 cc. of water colored with a crystal of potassium permanganate. *Is the distillate colorless? (Don't taste it.)*

b. Add a few drops of concentrated solution of ammonia (a gas) to 50 cc. of water. Note the odor of the liquid and place a drop of it on red litmus paper. *Result?*

Distill the dilute ammonia water in the same way in which you did the salt water. *What is the odor of the distilled liquid? Test it with red litmus paper. Is it free from ammonia? Explain.*

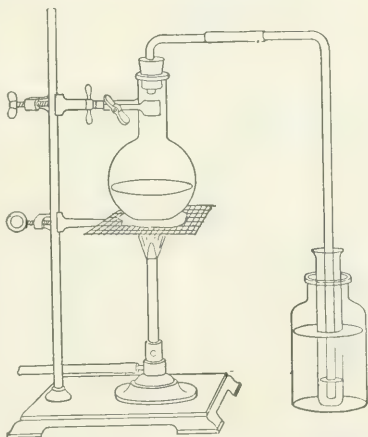


Fig. 28. Simple apparatus for distilling water.

**Optional work.** Set up a Liebig condenser as represented in figure 29. Use 100 cc. of ordinary water and add to it one drop of concentrated ammonium hydroxide solution. In each of six clean test tubes place one drop of phenolphthalein solution (an

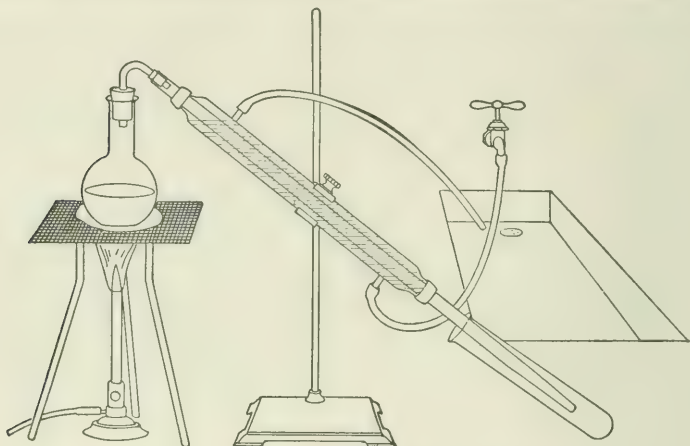


Fig. 29. Distilling water with Liebig condenser.

indicator which turns pink with ammonia) and catch the distillate in these test tubes. Change the test tube as soon as 15 cc. of liquid has come over. *Does the ammonia pass over with the steam? Do the first portions of the distillate contain more or less ammonia than the later portions?*

## EXPERIMENT 12

### CARBON DIOXIDE — PREPARATION AND PROPERTIES

**Apparatus.** — Wide-mouth bottle with 2-hole stopper. Thistle tube.

3 wide-mouth bottles and glass plates. Glass and rubber tubing.

**Materials.** — Marble chips. Dilute hydrochloric acid. Litmus paper (blue). Tapers. Limewater. Splint.

**a. Preparation.** Use the same apparatus which you used to generate hydrogen, and arrange to collect 3 bottles of gas by dis-

placement of air (Fig. 30). Slide into the bottle (don't drop them in) enough marble chips to cover the bottom. Add dilute



Fig. 30. Generating carbon dioxide and displacing the air.



Fig. 31. Shaking carbon dioxide with water.

hydrochloric acid through the thistle tube until a brisk evolution of gas is obtained. To test when a bottle is filled, bring a burning splint to its mouth; the gas will extinguish the flame. Cover each bottle with a glass plate. Allow the gas to bubble through a little water in a test tube. Taste the liquid and test it with litmus paper.

**b. Properties.** Light a taper and pour carbon dioxide from one of the bottles over it as you would pour water. *Does the gas support combustion? Is the gas heavier or lighter than air?*

**c.** Pour about 10 cc. of water into a bottle of the gas, cover at once tightly with the palm of the hand (Fig. 31), and shake it vigorously. *Is the hand sucked in, that is, held against the mouth of the bottle by atmospheric pressure? Is the gas soluble?*

**d.** Pour about half a test tube full of clear limewater into a bottle of carbon dioxide and shake the bottle. The milkiness



Fig. 32. Blowing through limewater.

is due to the calcium carbonate which is precipitated. This serves as a good test for carbon dioxide.

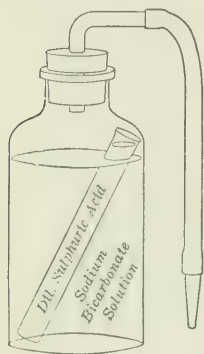


Fig. 33. Simple form of chemical fire extinguisher.

dilute sulphuric acid (about 1 to 10).

To show its operation, build a little fire of paper and splints in a pan and then grasp the bottle so as to hold the stopper in firmly and invert. Put out the fire. *Explain its operation.*

e. Burn a pine splint in a bottle of air. Add a little limewater and shake the bottle. *What gas is formed when wood burns?*

Blow through a glass tube into a test tube half full of limewater (Fig. 32). *What gas does the air exhaled from the lungs contain?*

f. Make a list of the properties of carbon dioxide which you have observed.

**Optional work.** The construction and operation of one form of fire extinguisher may be shown by the apparatus in figure 33. The bottle is nearly filled with a saturated solution of sodium bicarbonate (baking soda), and the small test tube with very

## EXPERIMENT 13

### CARBON MONOXIDE — PREPARATION AND PROPERTIES

**Apparatus.** — Ring stand with 1 ring and clamp. Wire gauze with asbestos center. Bunsen burner. Small flask with 2-hole stopper fitted with delivery tube and dropping funnel. Pneumatic trough. 3 wide-mouth bottles and glass squares.

**Materials.** — Concentrated sulphuric acid. Formic acid. Taper. Limewater.

**CAUTION.** Carbon monoxide is poisonous. Do not inhale it.

a. **Preparation.** Fit a small flask with a two-hole stopper carrying a delivery tube and a funnel connected by a short

rubber tube and pinchcock with a straight tube as shown in figure 34. Set up the apparatus on a ring stand with the asbestos wire netting under the flask at such a height as to heat the flask very gently with a small flame. Arrange the delivery tube for collecting the gas over water. Remove the stopper from the flask and pour in 15 cc. of concentrated sulphuric acid. Replace the stopper and partially fill the funnel with formic acid. Never let this run out entirely during the experiment.

Allow a few drops of the formic acid to flow into the flask and warm very gently. Adjust the pinchcock so as to admit the formic acid one drop at a time and so as to get a steady flow of gas from the flask. Collect 3 bottles full of the gas and close the pinchcock to stop the generation of gas.

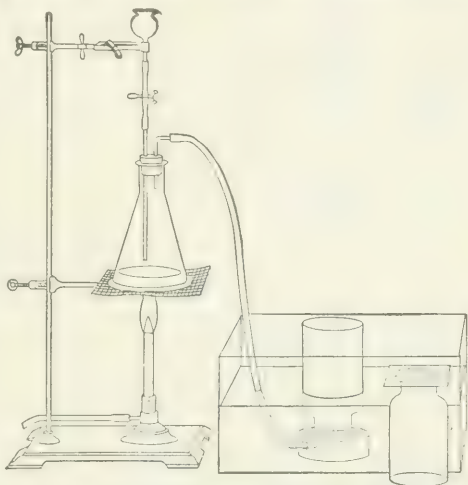


Fig. 34. Generating carbon monoxide by dropping formic acid on warm concentrated sulphuric acid.

**b. Properties.** Do not use the first bottle. *Why not?* Light a short taper and lift one bottle up mouth downward. Plunge the burning taper up into it. *Does the gas burn? Does the taper burn in the gas?*

**c.** Take another bottle of the gas and slip the glass plate partly off in order to pour into the bottle 5 cc. of clear limewater; then quickly replace the plate and, holding it tightly against the mouth of the bottle, shake the contents of the bottle. *Note any change in the limewater.* Now light a taper, remove the glass plate, and quickly ignite the gas. Replace

the cover at once and when the flame dies out, shake the contents again. What gas is formed when carbon monoxide burns?

**d. Summarize** the properties of carbon monoxide. How could you distinguish burning carbon monoxide from burning hydrogen?

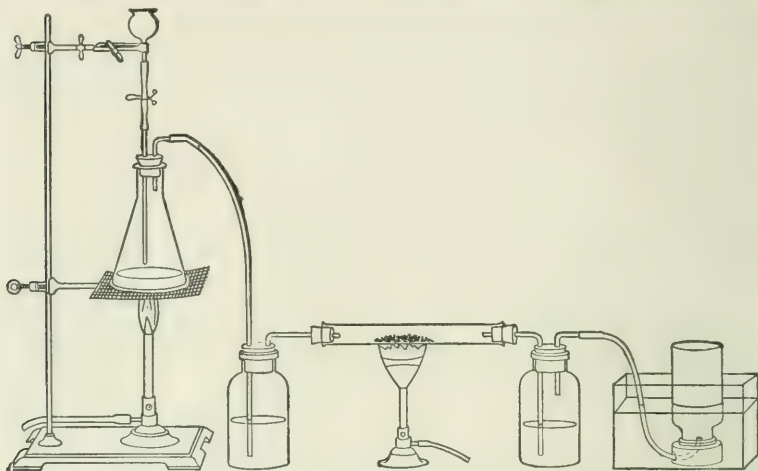


Fig. 35. Reducing copper oxide with carbon monoxide.

**Optional work.** The reducing action of carbon monoxide may be shown by passing the gas through water and then over hot copper oxide (Fig. 35). The carbon dioxide which is formed is shown by the milkiess produced in limewater. Any unchanged carbon monoxide is collected over water and then burned.

## EXPERIMENT 14

## ATMOSPHERIC NITROGEN

**Apparatus.** — A 2-liter bottle with 2-hole stopper. Hard-glass tubing (20 cm.) with two 1-hole stoppers. Bunsen burner. 3 wide-mouth bottles. Pneumatic trough. Ring stand and clamp. Tubing.

**Materials.** — Copper gauze or turnings.

**a. Preparation.** Since the oxygen of the air combines readily with hot copper and the nitrogen does not, it is easy to obtain nitrogen from air. Set up the apparatus as shown in figure 36.

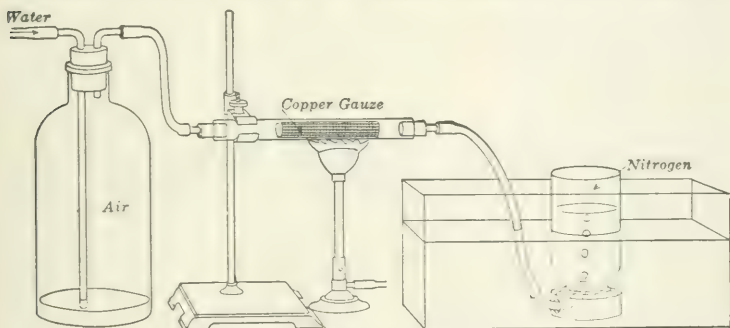


Fig. 36. Passing air over hot copper.

The hard-glass tube contains copper gauze or clean copper turnings. The current of air is obtained by letting water from the tap fill the bottle.

**b.** Heat the copper at first gently by moving the flame along the tube and then strongly. Do not burn the stopper. Carefully turn on the water so as to drive the air from the bottle very slowly over the hot copper. Collect three bottles of gas.

**c. Properties.** Apply a lighted taper to a bottle of the gas collected. *What happens?* Leave the bottle uncovered for 3 minutes and test again. *Does the taper now burn in the gas or not? Conclusions?*

Add a little clear limewater to another bottle and shake the contents. *Does the limewater turn milky?*

Find out whether burning sulphur will continue to burn in nitrogen.

*Make a list of the properties of nitrogen which you have observed.*

*What is the black substance seen on the copper after cooling?*

*How can you distinguish nitrogen from carbon dioxide?*

*Why was the air passed over the copper **slowly**?*

*What impurity does the nitrogen thus prepared contain? [§ 81]*

**Optional work.** By using copper and magnesium in a longer hard-glass tube (30 cm. long), it is possible to absorb both the oxygen

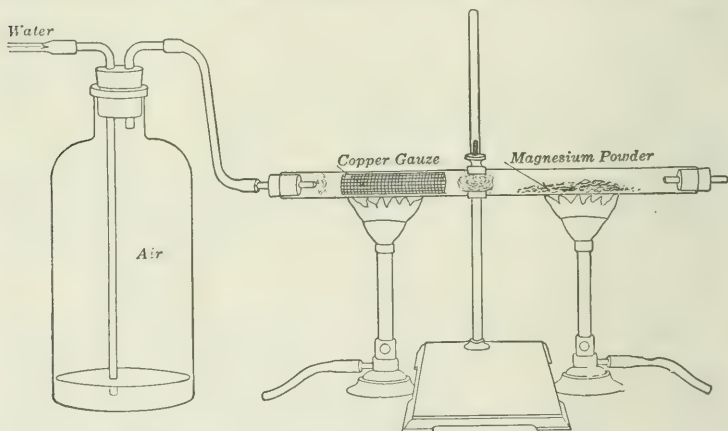


Fig. 37. Passing air over hot copper and magnesium.

and the nitrogen of air. Put a loose plug of shredded asbestos in the middle of the tube with copper gauze on the left side and magnesium powder on the right. Heat both metals as shown in figure 37 and then very slowly pass a steady stream of air through the tube from left to right. Continue as long as there is evidence of a change in both metals. Break the tube, *examine and name the products*. Place the magnesium product in water. *Note any odor.*

## EXPERIMENT 15

## PERCENTAGE OF OXYGEN IN AIR — VOLUMETRIC

**Apparatus.** — Pneumatic trough. Ring stand with clamp. 100 cc.-graduated cylinder. Thermometer. Barometer.

**Materials.** — White phosphorus. Copper wire (30 cm.).

a. A convenient method of determining what percentage of the air is oxygen consists in burning some substance in a definite volume of air and then measuring the volume of the remaining gas. White phosphorus is a good substance for this purpose because it forms an oxide which is soluble in water.

**CAUTION.** Great care must be taken never to handle white phosphorus except under water.

b. To get a definite volume of air invert a 100 cc.-graduated cylinder so that its mouth dips under the surface of the water in a pneumatic trough. The cylinder may be tipped to let part of the inclosed air escape, and then it should be securely clamped in a vertical position so that the water level inside and outside the cylinder is the same. *The volume of the air thus confined (about 100 cc.) should be carefully observed and recorded. Also record the barometric pressure and the temperature of the water, which should have stood long enough to be at the temperature of the room.*

c. A piece of ordinary (white) phosphorus should now be stuck on the end of a piece of copper wire (under water) and the wire carrying the phosphorus should be pushed up into the air space in the cylinder and allowed to stand overnight. It is well to arrange the apparatus (Fig. 38) so that the phosphorus does not touch the sides of the tube and stands well up toward the top of the cylinder.

Applying the Gas Laws, compute the volume that this air would occupy under standard conditions ( $0^{\circ}$  C. and 760 mm.).

d. The next day remove the phosphorus from the cylinder

and lower the cylinder until the level of the water inside is the same as that outside. Read and record the volume of the gas remaining in the cylinder, also the temperature of the water and the barometric pressure.

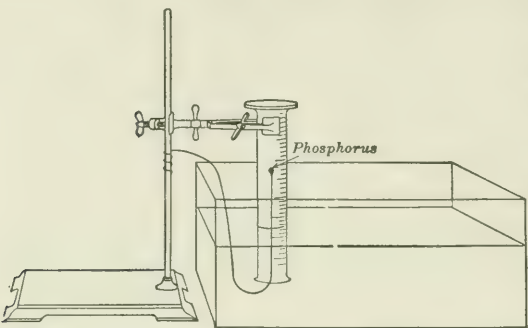


Fig. 38. Burning the oxygen out of air with phosphorus.

e. Compute the volume which the remaining gas would occupy if it were under standard conditions. Then calculate the difference between the original volume of air inclosed and the volume of gas that remains (both at standard conditions). This represents the volume of oxygen removed by the phosphorus.

Calculate the percentage of oxygen in air as follows:

Volume of air : Volume of oxygen :: 100 : x

f. What properties did you observe in the gases left after the oxygen had been absorbed? What were the gases?

Make all calculations in your notebook and record your observations and the results of your calculations in tabular form:

Volume of air taken	cc.
Temperature of the water, first day	°C.
Barometric pressure, first day	mm.
Volume of air at 0° C. and 760 mm.	cc.
Volume of gas remaining in cylinder	cc.
Temperature of water, second day	°C.
Barometric pressure, second day	mm.
Volume of gas remaining at 0° C. and 760 mm.	cc.
Volume of oxygen absorbed by phosphorus	cc.
Percentage of oxygen in air (by volume)	%

**Optional work.** Another quicker method of absorbing the oxygen from air is by means of an alkaline solution of pyrogallol. This method however, requires considerable dexterity to get good results.

Set up the apparatus as in figure 39. Disconnect the large test tube temporarily from the stopper. Mix 3 cc. of pyrogallie acid solution\* and pour this into the funnel. Now open the pinchcock slightly and permit the solution to fill the rubber and glass tubes completely down to the opening of the nozzle. Replace the test tube, fitting the stopper tightly into its mouth, and thus inclose a volume of air equal to the capacity of the test tube.

Now open the pinchcock. A little of the solution will be forced into the test tube by the pressure of the liquid above, and, as the oxygen is absorbed, more will enter. When the liquid stops flowing in, close the pinchcock and turn the test tube upside down several times to absorb the last traces of oxygen. Care must be taken not to warm the tube by handling.

Finally while the test tube is in the inverted position, reopen the pinchcock and equalize the levels of the liquid in test tube and funnel by raising or lowering the former. Then close the pinchcock, restore the test tube to its original position, and mark the positions of the bottom of the stopper and of the surface of the liquid by means of rubber bands.

Clean the apparatus and measure with a graduated cylinder the volume of the test tube up to the lower and upper bands. The former is the volume of the oxygen, and the latter that of the air. Calculate the *percentage of oxygen in air* (by volume), *i.e.* the number of cubic centimeters of oxygen in 100 cubic centimeters of air.

\* Use 5 g. pyrogallie acid in 15 cc. water and 120 g. sodium hydroxide in 80 cc. water.

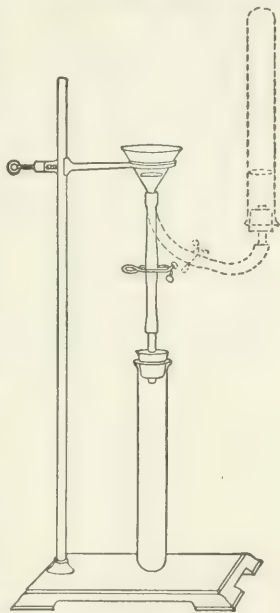


Fig. 39. Absorption of oxygen in the air by pyrogallol.

## EXPERIMENT 16

## HYDROCHLORIC ACID

**Apparatus.** — Ring stand with clamp. Bunsen burner. Asbestos gauze. Florence flask with 2-hole stopper. Thistle tube. Wide-mouth bottles and glass squares. Glass tubing. Cardboard. Dish. Stirring rod.

**Materials.** — Sodium chloride (common salt). Concentrated sulphuric acid. Litmus papers. Concentrated ammonium hydroxide. Splint. Filter paper. Magnesium.

**a. Preparation.** Arrange a flask with stopper, thistle tube, and delivery tube as shown in figure 40. Pour into the flask 7 cc. of water and then pour in slowly 20 cc. of concentrated sulphuric acid ( $\text{H}_2\text{SO}_4$ ).

**CAUTION.** Always pour the sulphuric acid into the water.

Cool the diluted acid by holding the flask in a stream of running water. When the acid is cold put into it about

15 g. of common salt ( $\text{NaCl}$ ). Replace the stopper carrying the thistle tube and delivery tube and set the flask on the asbestos gauze. Clamp the neck of the flask at the right height for heating with a Bunsen burner. The thistle tube must extend almost to the bottom of the flask so that its end may be under the liquid. Put the delivery tube through a piece of cardboard and let it extend

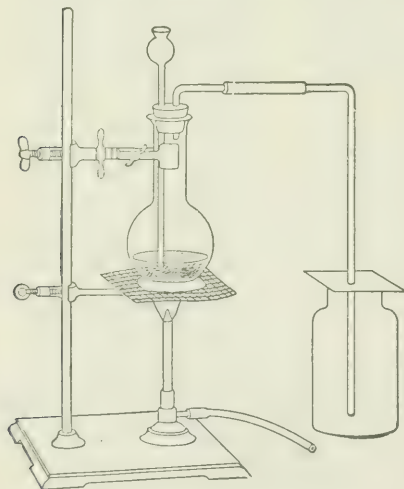


Fig. 40. Preparing hydrogen chloride.

nearly to the bottom of a dry clean bottle.

Heat the flask very slowly with a small flame. You can tell when the bottle is full of the hydrogen chloride gas by holding a strip of moist blue litmus paper at the mouth of the bottle. When the paper turns red, the bottle is full of the gas ( $\text{HCl}$ ) and should be replaced at once by another. Collect three bottles of gas and cover them with glass plates. Then fill another bottle about one-third full of distilled water and place the end of the delivery tube just below the surface of the water. Continue heating and observe the currents in the water at the end of the delivery tube. *What causes them?* Avoid letting the hydrogen chloride gas escape into the room.

**b. Properties.** Invert a bottle of the gas in a dish of water, remove the glass plate, and *note what happens*.

Test the gas in another bottle with a lighted splint. *Is it combustible? Is it a supporter of combustion?*

Pour a little ammonium hydroxide on a wad of filter paper and throw it into a bottle of hydrogen chloride. *What is the result?*

**c. Hydrochloric acid** is the water solution of hydrogen chloride. Dip a stirring rod into the solution and apply a drop to red and blue litmus papers.

Pour some of the hydrochloric acid just made into a test tube and drop into it a strip of magnesium. Test the gas set free with a flame. *Result? Write the equation for this reaction.*

**Optional work.** Place in separate tubes small quantities of the following chlorides: potassium chloride ( $\text{KCl}$ ), ammonium chloride ( $\text{NH}_4\text{Cl}$ ), calcium chloride



Fig. 41. Testing for hydrogen chloride with a drop of ammonium hydroxide.

( $\text{CaCl}_2$ ), and **ferric chloride** ( $\text{FeCl}_3$ ). Add a few drops of concentrated sulphuric acid ( $\text{H}_2\text{SO}_4$ ) and, if necessary, warm gently to start the reaction. *Is the material boiling?* Waft a little of the gas toward the nose. *What is the odor?* Blow across the mouth of each tube. *How does this gas behave with water vapor?* Place moistened blue litmus paper across the mouth of each test tube. *What does this show about the aqueous solution of the gas?* Dip a glass rod in ammonium hydroxide solution ( $\text{NH}_4\text{OH}$ ); now insert the glass rod into the mouth of the test tube (Fig. 41). The product is ammonium chloride ( $\text{NH}_4\text{Cl}$ ).

## EXPERIMENT 17

### TEST FOR A CHLORIDE

**Apparatus.** — Test tubes and rack.

**Materials.** — Dilute hydrochloric acid. Dilute nitric acid. Solutions of silver nitrate (5 %), ammonium hydroxide, sodium phosphate, and potassium oxalate. "Unknown" solution.

a. Fill a test tube half full of dilute hydrochloric acid and add a few drops of silver nitrate solution ( $\text{AgNO}_3$ ). The solid which separates out, *i.e.* the **precipitate**, is silver chloride ( $\text{AgCl}$ ).

*Record the color and appearance of this precipitate.* Label and set the tube aside for further experimenting.

b. In the same way add a few drops of silver nitrate solution to a solution of sodium phosphate. The precipitate in this case is silver phosphate.

*Record its color and appearance.* Label the test tube and set it aside.

c. To a solution of potassium oxalate add a few drops of silver nitrate solution. The precipitate is silver oxalate.

*Record its color and appearance.* Label this test tube and set it aside.

d. Try the effect of dilute nitric acid on each of the three precipitates just prepared. *How can you distinguish between silver chloride, silver phosphate, and silver oxalate?*

**e.** Prepare another portion of silver chloride by adding silver nitrate solution to a solution of sodium chloride (common salt). *Find out whether this precipitate is soluble in ammonium hydroxide.*

**f.** Prepare still another portion of silver chloride and stand the tube in the sunlight. *What is the result?*

*Summarize the three characteristic properties of silver chloride. How would you test for a soluble chloride?*

**g.** Apply these tests to an "unknown solution" which you may obtain from the instructor. In making your report on this unknown solution, record the result of each step in the process of testing as well as your final conclusion.

## EXPERIMENT 18

## ACIDS, BASES, AND SALTS

**Apparatus.** — Test tubes and rack. Stirring rod. Evaporating dish. Tripod and asbestos gauze. Glass plate. Bunsen burner.

**Materials.** — Dilute hydrochloric, sulphuric, nitric, and acetic acids. Litmus papers (red and blue). Solutions of sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. Sodium hydroxide (sticks).

**a. Acids.** Prepare dilute solutions of the following acids by adding a few drops of the acid to 10 cc. of water: hydrochloric, sulphuric, nitric, and acetic. Lay a piece of blue litmus paper and a piece of red litmus paper on a glass plate. Touch both kinds of paper with the end of a clean glass rod wet with each acid in turn. *What changes do you observe in each case?*

Taste one drop of each of the dilute solutions and immediately after tasting rinse the mouth with water. *What is the distinctive taste of acid?*

*Write the formulas of these acids (see textbook) and find out what element is present in all of them.*

**b. Bases.** In a similar way test with litmus papers dilute solutions of the following bases: sodium hydroxide, potassium hydroxide, ammonium hydroxide, and calcium hydroxide. *What is the characteristic change in litmus which you observe with bases?*

Taste a drop of calcium hydroxide solution. *How would you characterize the taste of bases?*

*Write out the formulas of these bases and find out in what respect they are similar in composition.*

**c. Salts.** Dissolve a small piece of sodium hydroxide (a stick about 1 cm. long) in about 20 cc. of water in a test tube. Stir it until all is dissolved and then pour out about 15 cc. of the solution into an evaporating dish (Fig. 42). Add dilute hydrochloric acid a little at a time and keep stirring the

liquid until a drop on a glass rod gives no basic reaction with litmus.

Then dilute the 5 cc. of sodium hydroxide solution which was left in the test tube until it is five times as dilute, and also dilute the acid solution the same amount. Now use these much diluted acid and base solutions to bring the solution in the evaporating dish to the neutral point, or **end point**, i.e. the point where it affects neither red nor blue litmus paper. This process is called **neutralization**.



Fig. 42. Neutralizing a base with an acid.

Evaporate the neutral solution to dryness slowly so that the liquid does not spatter. Taste the residue. *What is it?*

The scientific name for the residue is sodium chloride, which indicates that it contains only sodium and chlorine. *What has probably become of the other elements in the acid and base?*

**Optional work.** The process in part (c) is a general method of preparing neutral salts. Prepare other salts, such as potassium nitrate ( $\text{KNO}_3$ ), sodium sulphate ( $\text{Na}_2\text{SO}_4$ ), or ammonium chloride ( $\text{NH}_4\text{Cl}$ ).

## EXPERIMENT 19

## PREPARATION OF SOLUBLE SALTS

**Apparatus.** — Test tubes and rack. Tripod and asbestos gauze.

Evaporating dish. Bunsen burner. Funnel. Stirring rod.

**Materials.** — Dilute hydrochloric, nitric, and sulphuric acids. Zinc. Magnesium oxide. Sodium carbonate. Dilute solutions of sodium hydroxide and potassium hydroxide. Litmus papers. Filter paper.

**a. Metal and acid.** Many of the soluble salts (see Table of Solubilities in Appendix) of the common metals, such as zinc (Zn), iron (Fe), magnesium (Mg), and copper (Cu), can be prepared by replacing the hydrogen in the common acids. Such salts as zinc chloride ( $\text{ZnCl}_2$ ), magnesium sulphate ( $\text{MgSO}_4$ ), copper nitrate ( $\text{Cu}(\text{NO}_3)_2$ ), and iron sulphate ( $\text{FeSO}_4$ ) can be thus prepared.

To 5 cc. of dilute hydrochloric acid add zinc until there is no further action. Filter, evaporate the filtrate almost to dryness, and cool. *What is the residue? Write the equation.*

**b. Metallic oxide and acid.** Dissolve about 5 g. of magnesium oxide ( $\text{MgO}$ ) in dilute sulphuric acid and filter if necessary; then evaporate the clear solution to a small volume and cool. *What is the residue? Write the equation.*

**c. Carbonate and acid.** Heat 10 cc. of a solution of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) to the boiling point and add dilute hydrochloric acid as long as it causes effervescence. Evaporate the resulting solution to dryness. *What is the residue? Write the equation.*

Try the action of hydrochloric acid on solid sodium carbonate. *Compare the action of hydrochloric acid on a carbonate with the action of sulphuric acid on a chloride.*

**d. Neutralization of a base and an acid.** Sodium chloride ( $\text{NaCl}$ ) has already (Exp. 18) been prepared by neutralizing sodium hydroxide ( $\text{NaOH}$ ) with hydrochloric acid. In a similar

way potassium chloride ( $\text{KCl}$ ), sodium sulphate ( $\text{Na}_2\text{SO}_4$ ), potassium sulphate ( $\text{K}_2\text{SO}_4$ ), sodium nitrate ( $\text{NaNO}_3$ ), and potassium nitrate ( $\text{KNO}_3$ ) can be prepared.

Make one of these salts by neutralizing a suitable base and acid. Evaporate the salt solution nearly to dryness and examine the residue. *Write the equation.*

## EXPERIMENT 20

### PREPARATION OF INSOLUBLE SALTS

**Apparatus.** — Test tubes and rack. Small beakers. Asbestos gauze. Bunsen burner. Tripod.

**Materials.** — Solutions of lead nitrate and lead acetate. Lead oxide (litharge) and lead carbonate. Sodium chloride solution. Dilute hydrochloric acid.

We may illustrate the various ways of preparing an insoluble salt by preparing lead chloride ( $\text{PbCl}_2$ ) as follows:

**a. Salt and acid.** Heat in a test tube 20 cc. of lead nitrate ( $\text{Pb}(\text{NO}_3)_2$ ) solution to boiling and add 5 cc. of dilute hydrochloric acid. Set aside to cool. *What is the precipitate? Write the equation.*

**b. Salt and salt.** Heat 20 cc. of lead acetate ( $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ) solution and add 5 cc. of sodium chloride solution. Set aside to cool. *What is the precipitate? Write the equation.*

**c. Oxide and acid.** Dissolve in a beaker 1 g. of lead oxide ( $\text{PbO}$ ) in boiling dilute hydrochloric acid and add about 50 cc. of boiling water. Decant the clear solution and set it aside. *What is the precipitate? Write the equation.*

**d. Salt and acid.** Dissolve 1 g. of lead carbonate ( $\text{PbCO}_3$ ) in boiling hydrochloric acid and proceed as in part (c).

**Optional work.** Devise methods of preparing barium sulphate ( $\text{BaSO}_4$ ), lead sulphate ( $\text{PbSO}_4$ ), silver chloride ( $\text{AgCl}$ ), and lead iodide ( $\text{PbI}_2$ ). Prepare one of these salts in as many ways as your time permits and submit samples. Find out how this salt is prepared commercially and why.

## EXPERIMENT 21

## SOLUTIONS OF GASES, LIQUIDS, AND SOLIDS

**Apparatus.** — Test tubes and rack. Bunsen burner. Funnel. Watch glasses.

**Materials.** — Concentrated ammonium hydroxide. Carbon tetrachloride. Glycerin. Copper sulphate. Potassium chlorate. Calcium carbonate (powdered). Filter papers.

**a. Gases in water.** Warm gently in a test tube a little water drawn from the faucet. *Is there any evidence of a gas having been dissolved in the water?*

In the same way heat gently a little ammonia water (ammonium hydroxide) and note any evidence of a gas having been dissolved in the water.

*Are gases more soluble in hot or in cold water?*

**b. Liquids in liquids.** Add to 5 cc. of water in a test tube 5 cc. of carbon tetrachloride, a few drops at a time, and shake the mixture vigorously after each addition. *Are water and carbon tetrachloride miscible at each stage?*

Repeat, using glycerin and water.

**c. Solids in liquids.** Place in separate test tubes about 1 g. of each of the following powdered substances: copper sulphate ( $\text{CuSO}_4$ ), potassium chlorate ( $\text{KClO}_3$ ), and calcium carbonate ( $\text{CaCO}_3$ ). Add to each substance 10 cc. of water and shake each test tube.

*What evidence is there that these substances have dissolved?*

If there is any doubt as to whether any of a solid has dissolved, filter off 5 cc. of the liquid and evaporate on a watch glass to dryness.

If in any case all the solid has dissolved, add another gram of the powdered solid and continue this process as long as it is completely dissolved.

*What are the relative solubilities of these three substances in water?*

**Optional work.** What are the best conditions for the solution of a solid? Study the **effect of surface**. Take two crystals of copper sulphate of about the same size, each of which can be slipped into a test tube. Grind one of the crystals to a fine powder in a mortar and place the powder in a test tube. In another test tube place the other crystal. Add 20 cc. of water to each test tube; cork and shake the tubes gently. Determine how long it takes the powder to dissolve and what fraction of the original crystal still remains in the tube. *Why does pulverizing hasten the process of solution?*

## EXPERIMENT 22

### EFFECT OF TEMPERATURE ON SOLUBILITY

**Apparatus.** — Test tubes and rack. Ring stand. Bunsen burner. Asbestos gauze. Large beaker.

**Materials.** — Copper sulphate. Sodium chloride.

**a. Solubility in cold water.** Add to 10 cc. of water in a test tube 2 g. of well-pulverized copper sulphate and shake the tube until the solid dissolves. Continue adding 2-gram portions until the solution is saturated, that is, until some of the solid remains undissolved after shaking. *Calculate the amount (g.) which would dissolve in 100 g. of cold water.*

Repeat the experiment, using sodium chloride (common salt) instead of copper sulphate.

**b. Solubility in hot water.** Heat the solutions just prepared in a beaker of boiling water (Fig. 43) and continue to add 2-gram portions of the solid until each solution is saturated at  $100^{\circ}\text{C}$ . *Compute for each solid the amount which dissolves in 100 g. of hot water.*

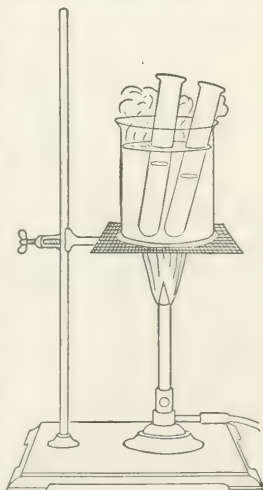


Fig. 43. Heating solutions in hot water.

**c. Cooling a saturated solution.** Cool the solutions just prepared (b) by holding the test tubes in running water from the faucet. *Observe the amount and size of the crystals formed in each tube.*

Warm the contents of the tubes carefully by putting them once more into the boiling water until the crystals are again dissolved. Set the tubes away in the test-tube rack and let them cool slowly until the next laboratory period. *Compare the crystals formed quietly and slowly with those formed quickly.*

**Optional work. Supersaturation.** Make a saturated solution of sodium sulphate crystals by shaking the salt with water at  $33^{\circ}\text{C}$ . Reheat the solution if it falls below that point (**transition temperature**). Pour into three test tubes clear portions of the solution. Cool the tubes in cold water without shaking. The solutions should now contain no crystals.

Put into one portion a very small crystal of the salt; into another portion a bit of sodium sulphate which has been dehydrated by heating on a glass rod; and shake the third portion violently.

*Compare the results of these experiments.*

## EXPERIMENT 23

### SOLUBILITY OF COMMON SALT

*To find how many grams of common salt will dissolve in 100 grams of water.*

**Apparatus.**—Small flask with stopper. Evaporating dish. Horn pan balance and weights. Ring stand. Copper water bath or large beaker. Tripod. Bunsen burner. Thermometer. Funnel.

**Materials.**—Sodium chloride. Filter paper.

**a.** First make a saturated solution of sodium chloride. This can be done by placing 10 g. of common salt with 20 cc. of water in a stoppered flask or bottle and shaking the contents at intervals for 15 minutes; or by dissolving 10 g. of salt in 20 cc. of boiling water and allowing the solution to cool to the

temperature of the room (shaking the solution occasionally). In either case some of the salt should remain undissolved.

**b.** Take the temperature of the saturated salt solution just prepared. Carefully weigh a clean, dry evaporating dish and then pour into it through a filter a portion of the solution. Reweigh as rapidly as possible, but carefully.

**c.** Evaporate the solution completely to dryness upon a water

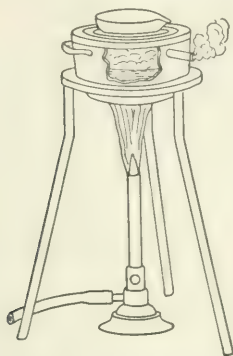


Fig. 44. Evaporating over a water bath (copper).

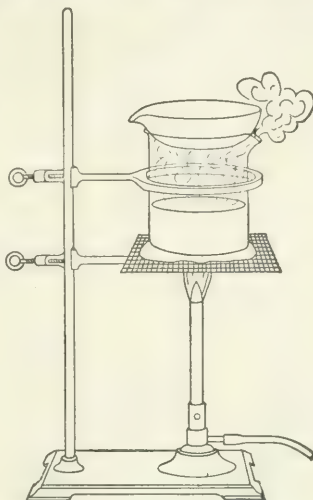


Fig. 45. Evaporating on a beaker of boiling water.

bath (Fig. 44) or on a beaker of boiling water (Fig. 45). Be careful not to lose any of the salt by spurting. Let the dish and contents cool and again weigh. Reheat the dish and contents until the weight is constant.

**NOTE.** Care must be taken to replace from time to time the water in the copper bath or beaker.

**d.** Compute the number of grams of salt soluble in 100 g. of water at the observed temperature. Compare your result with the results plotted in the curves shown in figure 86 on page 152 of the textbook. Record your data and results in tabular form somewhat as follows:

Temperature of solution . . . . .	C.
Wt. of dish + solution . . . . .	g.
Wt. of dish . . . . .	g.
Wt. of solution . . . . .	g.
Wt. of dish + dry solid . . . . .	g.
Wt. of dish . . . . .	g.
Wt. of salt . . . . .	g.
Wt. of water . . . . .	g.
Wt. of salt dissolved in 100 g. of water at °C. . . . .	g.

**Optional work.** The same method can be applied to determine the solubility of copper sulphate ( $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ ) or potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) at various temperatures.

## EXPERIMENT 24

### WATER OF CRYSTALLIZATION — EFFLORESCENCE — DELIQUESCENT

**Apparatus.** — Three watch glasses. Ring stand with clamp. Bunsen burner. Test tubes.

**Materials.** — Crystals of sodium carbonate and gypsum. Fused calcium chloride. Crystals of potassium chlorate, potassium nitrate, potassium alum, zinc sulphate, sodium sulphate, and copper sulphate.

**a.** Place a fresh glassy crystal of washing soda (sodium carbonate) on a watch glass and set it aside until the next laboratory period. Treat in the same way a small crystal of gypsum and a piece of fused calcium chloride. *After a day or two describe any changes which you observe in the appearance of each substance.*

**NOTE.** Sodium carbonate crystals are **efflorescent** in air which contains the average amount of moisture.

Calcium chloride is **deliquescent** under the same conditions.

**b.** Place a clear crystal of washing soda in a dry test tube and clamp the tube in a horizontal position. Heat the crystal gently and observe any changes in its appearance. *What condenses on the walls of the tube?*

c. In separate dry test tubes warm gently (Fig. 46) a few crystals of each of the following substances: potassium chlorate, potassium nitrate, potassium alum, zinc sulphate, and sodium sulphate.

*Record the results in tabular form as follows:*

SUBSTANCE HEATED	AMOUNT OF CONDENSED WATER	APPEARANCE OF RESIDUE

NOTE. The amount of condensed water may be indicated by the words, "a trace," "little," or "much." Even anhydrous substances often contain a little moisture, which is said to be "water mechanically held."

*Are all crystalline substances hydrates?*

d. In another test tube heat gently a small crystal of copper sulphate (blue vitriol). *Observe the change in color and form.*

When the tube is cold add a few drops of hot water. *Note the change in color. Pour the solution on a watch glass and set it aside to recrystallize. Compare the product with the original substance.*

**Optional work.** Test a small crystal of cobalt chloride (about 5 mm. across) in the same way that you did copper sulphate in (d). The solution is called "**sympathetic ink.**" Use a clean pen to write with cobalt chloride solution on paper. The writing while slightly moist is almost invisible, but it comes out when dried over a low flame.

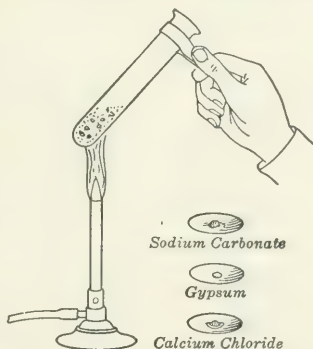


Fig. 46. Testing substances for water of crystallization.

## EXPERIMENT 25

## MEASUREMENT OF WATER OF CRYSTALLIZATION

*To find the per cent of water of hydration in gypsum.*

**Apparatus.** — Tripod. Pipe-stem triangle. Porcelain crucible.

Horn pan balance and weights. Bunsen burner. Ring stand.

**Materials.** — Gypsum crystals.

**a.** The amount of crystal water contained in a hydrate, such as gypsum, is found by heating a known weight of the crystallized substance until all the water is driven off. The process is completed when the substance ceases to lose weight on heating.

**b.** Weigh a clean, dry crucible with cover to the nearest centigram (0.01 g.). Place about 5 g. of pulverized gypsum (do not try to take this exact amount) in the crucible and weigh again with cover carefully. Record these weights in tabular form as shown below.

**c.** Place the covered crucible on the pipe-stem triangle and place this on the tripod. Heat the crucible at first very gently, holding the burner in the hand; then place the burner under the crucible and adjust the flame so that the top of the inner blue cone is a short distance below the crucible. After heating for 10 minutes, remove the cover and continue heating strongly for 5 minutes longer. Then *gradually* cool the crucible and reweigh to the nearest centigram.

**d.** Reheat the crucible (without cover) for 5 minutes longer, then allow to cool, and reweigh. Repeat this operation until the weight is constant. From your results calculate the percentage of water of hydration present in the crystals.

Wt. of crucible + gypsum . . . . .	g.
Wt. of crucible empty . . . . .	g.
Wt. of gypsum taken . . . . .	g.
Wt. after first heating . . . . .	g.

Wt. after last heating . . . . .	_____	g.
Wt. of water of hydration . . . . .	_____	g.
Percentage of water = $\frac{\text{wt. of water}}{\text{wt. of gypsum}}$ =		%

**Optional work.** Calculate from the results of this experiment the number of molecules of water in a molecule of gypsum. Assume the anhydrous substance is  $\text{CaSO}_4$  and use the approximate values for the atomic weights (Table facing front cover) to compute the molecular weights. Write the formula for gypsum.

## EXPERIMENT 26

## BOILING POINTS OF SOLUTIONS

**Apparatus.** — Ring stand with one ring and clamp. Asbestos gauze. Bunsen burner. Boiling flask (250 cc.) with side tube. Wide-mouth bottle. Sheet of cardboard. Thermometer.

**Materials.** — Alcohol. Salt solutions (10 g. in 100 cc. water and 20 g. in 100 cc. water). Sugar solution (20 g. in 100 cc. water).

**a. Water.** In order to show the effect of various substances on the boiling point of water, it is necessary first to determine

the boiling point of pure water. If the atmospheric pressure happened to be 760 mm., and if there were no error in the thermometer used, we should expect a thermometer placed in boiling water to indicate  $100^{\circ}$  on the Centigrade scale. But as it is hardly likely that both of these conditions obtain, we shall first arrange the apparatus as shown in figure 47 and then place a small quantity (4 or 5 cm. deep) of distilled water in the boiling flask. It is well

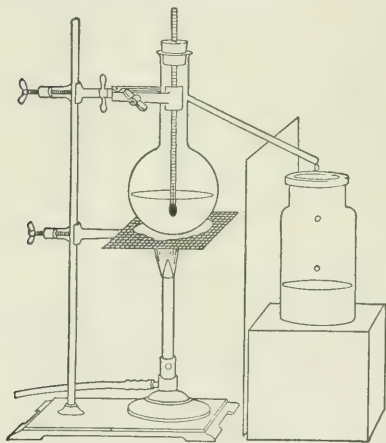


Fig. 47. Finding the boiling point of a solution.

to have the thermometer bulb just immersed in the water. When the Bunsen burner is placed underneath to boil the water, it should be adjusted so that the flame spreads on the asbestos pad in a spot about an inch in diameter.

Read and record the temperature as soon as steam issues freely from the exit-tube. The presence of some common glass beads in the flask will cause the water to boil more steadily

and will prevent "bumping." Continue to heat the water to see whether the temperature rises higher.

**b. Alcohol.** Fill the boiling flask half full of alcohol and determine its boiling point.

**CAUTION.** Alcohol vapor is inflammable. It is well to set up a cardboard between the flask and the delivery tube in order to avoid any possibility of igniting the escaping alcohol vapor with the flame.

**c. Alcohol and water.** Fill the boiling flask half full of a solution of alcohol and water (equal volumes of each) and determine its boiling point. *Does it remain constant as the liquid continues to boil? Compare the boiling point of this mixture with the boiling points of water and alcohol.*

**d. Salt and water.** In the same way determine the boiling point of a salt solution which contains 10 g. of common salt in 100 cc. of water. Then repeat, using a solution which contains 20 g. of salt in 100 cc. of water. Compute in each case the difference between the boiling point of the solution and of water. *What relation exists between these differences and the concentrations of the solution?*

**e. Sugar and water.** Determine the boiling point of a sugar solution which contains 20 g. of sugar in 100 cc. of water. *Compare the boiling point of the sugar solution with that of the salt solution of the same percentage concentration.*

Compute the molecular weight of common salt ( $\text{NaCl}$ ) and of common sugar ( $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ ). *Which contains the greater number of molecules, 20 g. of salt or 20 g. of sugar? Does this help to explain the difference in boiling points in the salt and sugar solutions?*

**f. Results.** *How can you tell whether a given liquid (a) is pure, (b) is a mixture of liquids, or (c) contains dissolved solids?*

**Optional work.** Repeat these experiments with the thermometer raised higher in the stopper so that the bulb is just below the side tube. This gives the temperature of the vapor of the boiling liquid. Compare the temperatures of the vapor of the boiling liquids with the temperatures of the liquids. Explain any differences.

## EXPERIMENT 27

## FREEZING POINTS OF SOLUTIONS

**Apparatus.** — Pint fruit jar or small battery jar. Test tubes. Thermometer.

**Materials.** — Cracked ice or snow. Rock salt. Salt solutions (10 g. in 100 cc. water and 20 g. in 100 cc. water). Sugar solution (20 g. in 100 cc. water).

**a. Freezing mixture.** Make a freezing mixture by filling a pint fruit jar, or a jar of about the same size, with a mixture of cracked ice and salt (about 3 to 1). Make a hole in the center so that a test tube may stand with its lower part surrounded by the freezing mixture (Fig. 48).

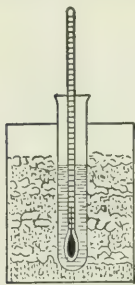


Fig. 48. Finding the freezing point of a solution.

**b. Water.** Pour some distilled water into the test tube (about an inch in depth), place it in position, and stir with the thermometer. From time to time raise the test tube a little and examine the contents. The ice will separate as flaky crystals. When it begins to do so, lift the tube from the freezing mixture, stir, and read the thermometer. Wait until the ice just begins to melt perceptibly; then stir and take another reading. If the two readings do not agree, find the average and call this the freezing point.

**c. Solutions.** In this way determine the freezing points of the following solutions:

Solution I — 10 g. common salt in 100 cc. water;

Solution II — 20 g. common salt in 100 cc. water;

Solution III — 20 g. common sugar in 100 cc. water.

**d. Results.** Calculate in each case the difference between the freezing point of water and that of the solution. *Does salt seem to lower or raise the freezing point of water? What is the*

*effect of varying the concentration of the salt solution? Why does the sugar solution produce so much less effect on the freezing point than the salt solution?*

**Optional work.** Experiments show that in the case of nonelectrolytes one gram molecule of a substance dissolved in 1000 cc. of water lowers the freezing point from  $0^{\circ}\text{C.}$  to  $-1.86^{\circ}\text{C.}$  Compute the gram-molecular concentration of the salt solutions used and then the depression of the freezing point as if it were not ionized at all. Finally, compute the degree of ionization, *i.e.* the per cent of the molecules which are ionized.

## EXPERIMENT 28

### FORMS OF SULPHUR

**Apparatus.** — Test tubes and rack. Crystallizing dish. Bunsen burner. Magnifying glass. Dish of water. Clamp. Funnel. Beaker.

**Materials.** — Roll sulphur. Carbon disulphide. Filter paper (10 cm. in diameter). Dime or other piece of silver.

**a. Crystals obtained from solution.** Put about 2 or 3 g. of powdered roll sulphur in a test tube, add about a quarter of a test-tubeful of carbon disulphide, and shake the tube.

**CAUTION.** Carbon disulphide is a volatile liquid which easily takes fire. No flame should be near it.

Pour off the clear solution (Fig. 49 A) into a crystallizing dish. Set it aside to evaporate the liquid at some distance from a flame.

(While waiting for the crystals to form, proceed with part b.)

When crystals have formed, examine them with a magnifying glass and note their shape; put them in a dry test tube and label them **rhombic sulphur**. *Draw several crystals in your notebook.*

**b. Crystals obtained by slowly cooling molten sulphur.** Place a folded filter paper in a funnel and have a dish of water at hand.

Fill a clean, dry test tube half full of powdered sulphur. Melt the sulphur very slowly by holding the test tube in an inclined position well above the flame (Fig. 49 B) and by turning the tube so as not to overheat the sulphur at any point. The liquid sulphur should be pale yellow; if it becomes dark in color, it shows that it has been overheated.

Pour the liquid sulphur into a folded filter paper (Fig. 49 c) and watch the crystals form. As soon as they extend from the sides to the center of the cone, break the crust and pour the

sulphur which is still liquid into the water in the dish. At once open up the filter paper and examine the crystals with a lens. *Make a drawing of a few of them and record their color and transparency.* This form of sulphur is called **prismatic sulphur**.

Keep some of these crystals and observe any change in their appearance.



FIG. 49 A.



FIG. 49 B.



FIG. 49 C.

Fig. 49. A, Pouring a solution of sulphur into a crystallizing dish. B, Melting sulphur in a test tube. C, Pouring melted sulphur into a filter paper.

**c. Plastic sulphur, obtained by heating sulphur to its boiling point and then suddenly cooling it.** Fill a beaker with water and place it on the table beside you. Half fill a test tube (preferably the one used in b) with small pieces of roll sulphur, hold the test tube in some kind of clamp (a folded strip of paper will serve), and very gently heat the sulphur until it melts. When it is completely melted, heat it more strongly and observe the changes in color and fluidity.

When the sulphur is boiling vigorously, pour it into the beaker of water (Fig. 50).

**CAUTION.** Do not become alarmed if the sulphur vapor begins to burn at the mouth of the test tube; do not, however, spill burning sulphur on the table.

*Observe what happens to the sulphur which remains in the test tube as it slowly cools.*

Take the solid from the beaker and examine it. *Note its color; is it hard or soft? is it elastic or brittle?* This form of sulphur is called **plastic sulphur** or **amorphous sulphur**.

Find out whether plastic sulphur is soluble or insoluble in carbon disulphide.

**d. Test for free sulphur.** Sulphur combines very readily with silver to form a black sulphide. Heat a minute particle of sulphur on a silver coin; a black stain is produced.

**Optional work.** Devise a suitable method for determining the **densities** of roll sulphur and plastic sulphur. If your method is approved by the instructor, carry it out.

Determine the **melting points** of rhombic sulphur and prismatic sulphur. Put small fragments of the two forms of crystals into separate melting-point tubes and attach them with rubber bands to the bulb of a thermometer, as shown in figure 51. Clamp the thermometer so that its bulb dips into some olive oil in a beaker. Heat gently and stir the oil constantly until the first sign of melting occurs. *Record the temperature.*

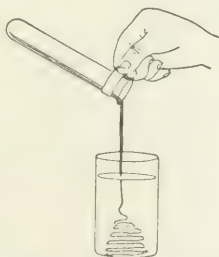


Fig. 50. Pouring boiling sulphur into cold water.

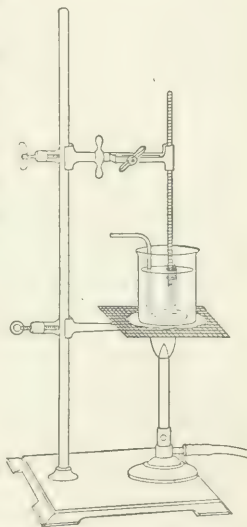


Fig. 51. Determining the melting point of sulphur.

## EXPERIMENT 29

## HYDROGEN SULPHIDE

**Apparatus.** — Ring stand and clamp. Large test tube ( $20 \times 2.5$  cm.) and 1-hole stopper to fit. Test tubes and rack. Bunsen burner. Glass tubing. Wide-mouth bottle. Glass nozzle.

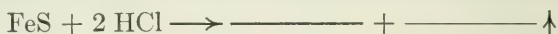
**Materials.** — Iron sulphide. Dilute hydrochloric acid. Solutions of lead nitrate, copper sulphate, and cadmium nitrate. Litmus papers.

**a. Preparation.** When iron filings and powdered sulphur are mixed together and heated, the product is iron sulphide (ferrous sulphide). Carefully slide two or three pieces of iron sulphide ( $\text{FeS}$ ) into a large test tube and pour in enough dilute hydrochloric acid to cover the iron sulphide.

*Note the odor of the gas.* Do not inhale much of it as it is somewhat poisonous.

**b. Tests.** The odor of the gas is a sufficient test when considerable quantities of the gas are present; but a more sensitive test is to dip a strip of filter paper into a solution of lead nitrate and to hold it in the gas to be tested.

Complete the equation for preparing hydrogen sulphide ( $\text{H}_2\text{S}$ ):



Also complete the equation for testing for the gas with lead nitrate:



**c. Properties.** Fit the test tube with a 1-hole stopper and delivery tube as shown in figure 52. Collect the gas in a dry test tube by the upward displacement of air, remove the delivery tube, and light the gas in this test tube. As soon as the gas in the test tube burns quietly, attach a glass nozzle\* or jet to the delivery tube and set fire to the gas.

\* A glass nozzle or jet is made by heating a straight piece of glass tube in the Bunsen flame and when soft, pulling it out so that the bore is reduced. Then cut the tube off at the constricted portion.

Cautiously smell the gas around the flame. Hold a cold, dry bottle over the flame and note whether any product of the combustion is condensed therein. *What are products of the complete combustion of hydrogen sulphide? Write the equation.*

Extinguish the flame and insert the delivery tube to the bottom of a test tube which is three-fourths full of water. Let the gas bubble through the water 3 or 4 minutes. (If the action in the generator slackens, gently warm the large test tube.) *What effect has this solution on blue litmus paper?* This solution is called **hydrosulphuric acid**.

**d. Uses.** A solution of hydrogen sulphide is a very useful reagent in chemical analysis to determine what metals are present in a material of unknown composition. Some of the metallic sulphides are insoluble and have distinctive colors.

Add a portion of the hydrosulphuric acid just prepared to a solution of copper sulphate. *What are the products formed? Complete the equation:*



Add another portion of the hydrosulphuric acid to a solution of cadmium nitrate. *What are the products formed? Complete the equation:*



**e. Summarize the observed properties of hydrogen sulphide.**

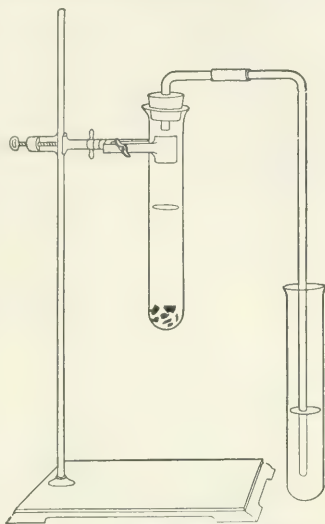


Fig. 52. Preparing a solution of hydrogen sulphide.

**Optional work.** Arrange six test tubes in a rack and fill each about one-third full of one of the following solutions: **zinc sulphate** ( $\text{ZnSO}_4$ ), **tin chloride** ( $\text{SnCl}_2$ ), **magnesium chloride** ( $\text{MgCl}_2$ ), **sodium chloride** ( $\text{NaCl}$ ), **antimony chloride** ( $\text{SbCl}_3$ ), and **lead nitrate** ( $\text{Pb}(\text{NO}_3)_2$ ). Bubble hydrogen sulphide through each solution in turn. *Note whether a precipitate is formed or not.*



**Fig. 53.** Bulb tube used to heat a sulphide in.

If no precipitate is formed, add a little ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) and *note any change*. If a precipitate is formed, filter, wash, dry, and heat in a bulb tube (Fig. 53) to find out whether the solid is a sulphide.

### EXPERIMENT 30

#### SULPHUR DIOXIDE AND SULPHUROUS ACID

**Apparatus.** — Platinum wire. Bunsen burner. Bulb tube. Ring stand with ring and clamp. Asbestos gauze. Erlenmeyer flask (250 cc.) with 2-hole stopper. Funnel. Pinchcock. Rubber connection tube. 3 wide-mouth bottles and glass squares. Test tubes. Evaporating dish. Glass tubing.

**Materials.** — Sulphur. Iron pyrite. Sodium bisulphite. Dilute hydrochloric acid. Pink flower. Thin slice of apple. Litmus papers. Zinc. Lead nitrate test paper.

**a. Preparation.** Touch a bit of sulphur with a warm platinum wire and bring the wire with adhering sulphur again into the flame. Withdraw it and observe the color of the flame of burning sulphur; also note the odor of the gas produced. *Write the equation.*

Heat (or roast) in a little bulb tube (Fig. 54) a few particles of iron pyrite ( $\text{FeS}_2$ ). *What is the solid which condenses on the cool part of the tube?*

A convenient method of preparing sulphur dioxide in the laboratory is to drop hydrochloric acid slowly on sodium hydrogen sulphite ( $\text{NaHSO}_3$ ). This reaction may be expressed by the following equation:



Arrange the apparatus as shown in figure 55. Place in the flask about 10 g. of sodium bisulphite (sodium hydrogen sulphite) and replace the stopper carrying the funnel tube and delivery tube. Attach a straight glass tube to the delivery tube in order to make it reach down to the bottom of a wide-mouth bottle.

Fill up the funnel with dilute hydrochloric acid and then adjust the pinchcock so as to admit the acid into the flask drop by drop. To increase the speed of the reaction, warm the flask *very gently* from time to time as needed.



Fig. 54. Heating iron pyrite in a bulb tube.

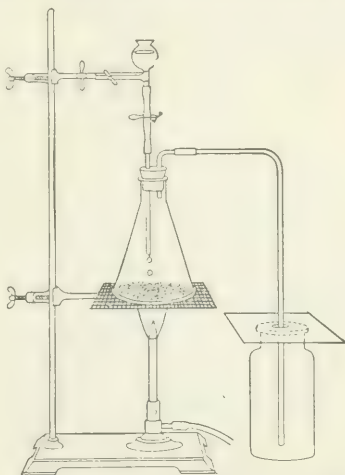


Fig. 55. Dropping hydrochloric acid on sodium bisulphite to generate sulphur dioxide.

As soon as the air in the flask has been swept out, collect two bottles of the gas by upward displacement of air. Cover each bottle with a glass plate. Boil half a test-tubeful of water so as to drive off the dissolved air and then cool under running water. Insert the delivery tube to the bottom of the boiled water and let the gas bubble through for 5 minutes. Cork this test tube just as soon as the delivery tube is taken out in order to keep out the air.

**b. Properties.** Uncover one of the bottles of gas just enough to pour in a little water; then close it *tightly* with the palm of the hand and shake the contents (Fig. 56). Is the hand held against the mouth of the bottle by atmospheric pressure (suction) or not? *Is the gas soluble?*

In the second bottle of gas place a moist pink flower (carnation) and a thin slice of apple. Leave another slice of apple exposed to the air outside. Compare the pieces. *What is the effect of sulphur dioxide on vegetable coloring matter?*



Fig. 56. Shaking a bottle containing sulphur dioxide and water.

**c. Sulphurous acid.** Test the aqueous solution made in part (a) with litmus papers. Write the equation for the formation of sulphurous acid ( $\text{H}_2\text{SO}_3$ ). *Is sulphur dioxide an acidic or a basic oxide?*

Boil 5 cc. of this solution slowly in an evaporating dish and note the odor from time to time. Write the equation which represents this change and compare it with the preceding equation.

To detect sulphurous acid, we may reduce it to hydrogen sulphide by using a suitable reducing agent, such as nascent hydrogen. Put a few pieces of zinc in a test tube and cover the metal with dilute hydrochloric acid. As soon as the action becomes vigorous, test the gas with a strip of moist lead nitrate paper (the paper should remain white). Now pour in 2 or 3 cc. of sulphurous acid and test again with lead nitrate paper.

**d. Summarize the properties of sulphur dioxide:** color, odor, density as compared with air, and solubility. Also **summarize the properties of sulphurous acid:** action on vegetable coloring matter, stability, oxidizing or reducing agent.

**Optional work.** Sulphurous acid and its salts, the sulphites, are often used as **food preservatives**, even though their use is questionable. **To test for sulphurous acid or sulphites in foods**, apply the method of part (c) and reduce the sulphurous acid, if present, to hy-

drogen sulphide. Examine one or more of the following: molasses, lime juice, mushrooms, Hamburg steak, sausage, catsups, fruit juices, and dried fruits.

Put about 25 g. of the sample (macerated if a solid) into a small Erlenmeyer flask (200 cc.). Add water, if necessary, to make a thin paste. Introduce about 5 g. of sulphur-free zinc and about 15 cc. of chemically pure concentrated hydrochloric acid ( $\text{HCl}$ ). Over the mouth of the flask place a small filter paper which has been moistened with a strong solution of lead nitrate ( $\text{Pb}(\text{NO}_3)_2$ ). Warm the solution gently. Blackening of the filter paper (not merely browning) indicates the presence of sulphites.

## EXPERIMENT 31

### SULPHURIC ACID

**Apparatus.** — Test tubes and rack. Bunsen burner.

**Materials.** — Concentrated sulphuric acid. Zinc. Dilute sulphuric acid. Sodium chloride. Wooden splint. Dilute solutions of sodium sulphate, zinc sulphate, and copper sulphate. Barium chloride solution. Dilute hydrochloric acid.

**a. Heat of dilution.** Fill a test tube about one-third full of water and then pour into it very gradually an equal volume of concentrated sulphuric acid. Touch the outside of the test tube and record any change in temperature.

**CAUTION.** Remember that the concentrated sulphuric acid is to be poured slowly *into* the water. The reverse order produces dangerously explosive spattering.

**b. Action on zinc.** Place a strip of zinc in a test tube and cover it with concentrated sulphuric acid. If there is no action, warm the acid cautiously.

**CAUTION.** Hot concentrated sulphuric acid causes very severe burns when it touches the skin. Do not pour hot concentrated sulphuric acid into water. Let it cool.

Place another strip of zinc in a test tube and cover it with dilute sulphuric acid. *Name the products formed in this reaction and write the equation.*

*Compare the action of concentrated and dilute sulphuric acid on zinc.*

**c. Action on salt.** Try the effect of both dilute and concentrated sulphuric acid on common salt (sodium chloride). *Identify the gaseous products.*

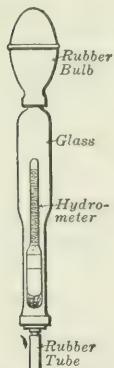
**d. Action on wood and paper.** Insert a splinter of wood into a little concentrated sulphuric acid in a test tube and let it stand for a few minutes.

Dip a glass rod into sulphuric acid and write with it on paper. Warm the paper gently over a flame. *What property of sulphuric acid do these experiments with wood and paper illustrate?*

**e. Test for a sulphate.** Prepare in separate test tubes dilute solutions of sulphuric acid, sodium sulphate, zinc sulphate, and copper sulphate. Add to each solution a few drops of barium chloride solution ( $\text{BaCl}_2$ ) and then dilute hydrochloric acid. *What insoluble precipitate is formed in each case?*

To a solution of sodium phosphate add a few drops of barium chloride and dilute hydrochloric acid. *Why is it necessary to add hydrochloric acid in testing for sulphates?*

*How would you distinguish between a sulphide, a sulphite, and a sulphate?*



**Fig. 57.** Syringe hydrometer for testing a storage battery.

**Optional work.** One of the most important differences in the physical properties of dilute and concentrated sulphuric acid is that of density. To determine the density of an acid take a small flask (25 cc.) and paste a label around the neck so that its upper edge is exactly horizontal. Find the weight of the flask empty and weigh the flask filled with water so that the lowest part of the water surface is exactly at the upper edge of the label. Then fill the flask to the same level with dilute sulphuric acid and find the weight. Still again find the weight when filled with concentrated sulphuric acid.

Calculate the density of these acids.

A very convenient method of testing a storage battery is to de-

termine the density of its electrolyte (dilute sulphuric acid) by means of a hydrometer (Fig. 57). Find out how this is done in a service station.

## EXPERIMENT 32

### AMMONIA — PREPARATION AND PROPERTIES

**Apparatus.** — Large test tube ( $20 \times 2.5$  cm.) fitted with 1-hole stopper.

Ring stand with ring and clamp. Bunsen burner. 3 wide-mouth bottles. Glass squares. Pneumatic trough. Glass tubing.

**Materials.** — Slaked lime. Ammonium chloride. Litmus papers.

Tapers. Concentrated hydrochloric acid.

**a. Preparation.** Ammonia ( $\text{NH}_3$ ) can be readily produced from an ammonium salt, such as sal ammoniac ( $\text{NH}_4\text{Cl}$ ), by heating it with any of the three common alkalis. Since lime is the cheapest alkali, it is the one commonly employed. As the gas, ammonia, is lighter than air, we may collect it in an inverted bottle by displacing the air.

Mix on a piece of paper 2 parts of powdered slaked lime ( $\text{Ca}(\text{OH})_2$ ) and 1 part of ammonium chloride ( $\text{NH}_4\text{Cl}$ ). Place this mixture in a large test tube which is provided with a delivery tube, as shown in figure 58. Do not fill the tube more than half full. Clamp it, incline slightly downward in order that any condensed moisture may not run back and crack the tube.

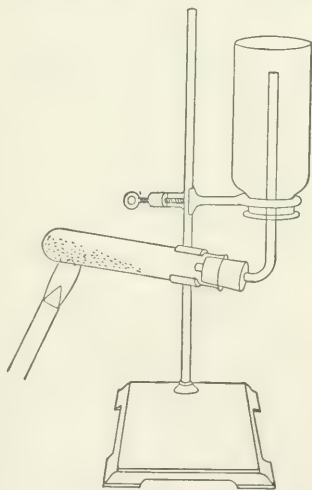


Fig. 58. Preparing ammonia and displacing the air.

Warm the mixture very gently, beginning with that portion near the mouth of the tube and gradually extending the heat to other portions, but keep the flame moving continually. As

soon as the smell of ammonia shows that the gas is escaping from the bottle, lift it up and cover its mouth with a glass plate. Set it aside mouth downward. In this way collect 3 bottles of the gas.

**b. Properties.** Put one bottle mouth downward into a basin of water (pneumatic trough). Remove the glass plate and shake the bottle a little. *Note what happens.* Replace the glass plate and remove the bottle with its contents. Test the liquid with litmus papers. *What is the action of ammonia on water? Write the equation.*

Pour into a warm, dry bottle two or three drops of concentrated hydrochloric acid. Cover this bottle with a glass plate and invert it upon a covered bottle of ammonia. Keep the bottles mouth to mouth while you remove both plates at the same time. The white solid which is formed is ammonium chloride ( $\text{NH}_4\text{Cl}$ ). *Write the equation for the reaction.*

Push a lighted taper up into an inverted bottle of the gas. *Does the gas burn? Does it support combustion of a taper?* Apply a lighted taper to the gas issuing from the exit-tube of the generator. *Observe whether the gas burns (1) while the lighted taper is present and (2) after it is removed.*

**c. Results.** Make a list of the properties of ammonia which you have observed in this experiment. Complete the equation for its preparation :



*How could you prove that a given substance was an ammonium salt?*

**Optional work.** To investigate other sources of ammonia, gently heat a mixture of gelatin and soda lime (sodium hydroxide and calcium hydroxide) in a test tube. Use very small equal amounts of these materials. *Note the odor of the gases given off and also their effect on moistened litmus paper.*

Smell ammonium sulphate and also slaked lime. Then rub these two substances together in the palms of the hands. *Note the odor and the effect on moistened litmus paper held over the mixture.*

Heat in a test tube a mixture of sodium hydroxide solution and ammonium nitrate. Test for ammonia with a drop of concentrated hydrochloric acid on a glass rod.

Investigate the dry "Ammo" powder. *What is its probable composition?*

### EXPERIMENT 33

#### NITRIC ACID

**Apparatus.** — Tubulated retort (250 cc.). Tripod. Bunsen burner. Asbestos gauze. Ring stand and clamp. Large test tube. Pneumatic trough. Funnel. Test tubes.

**Materials.** — Sodium nitrate. Concentrated sulphuric acid. Excelsior. White woolen yarn. Concentrated ammonium hydroxide. Copper turnings. Zinc. Ferrous sulphate solution, freshly prepared.

**CAUTION.** Nitric acid is very corrosive. Do not drop it on your flesh or clothes.

**a. Preparation.** Place about 30 g. of sodium nitrate (or potassium nitrate) on a piece of paper and fold the paper so that the solid may be slipped into the retort through the tubulure. Support the retort on the wire gauze with asbestos center, which is itself supported on a tripod. Clamp the neck of the retort in such a way (Fig. 59) that it extends into a large test tube or small flask partly immersed in cold water. Pour 20 cc. of concentrated sulphuric acid through a funnel upon the nitrate and then carefully remove the funnel without dropping acid from it. Replace the glass stopper in the tubulure.

As soon as the sulphuric acid has moistened the entire mass of nitrate crystals, begin heating the retort gently. Distill at

as low a temperature as possible until no more nitric acid condenses in the neck of the retort.

Allow the retort to cool in position. The residue, which is sodium (or potassium) hydrogen sulphate ( $\text{NaHSO}_4$ ), may be removed with warm water. *Write the equation for the preparation of nitric acid.*

**b. Properties.** The nitric acid just prepared is 100% pure, and great care must be used in handling it. Pour 1-2 cc. of this acid into a test tube and insert a small, loose wad of excelsior so that it remains about an inch above the acid.



Fig. 59. Making nitric acid with a tubulated retort.

Holding the test tube in a holder, boil the acid vigorously until the vapor reaches the excelsior. Now move the test tube so as to heat the excelsior itself for a few seconds. *How does this experiment illustrate the oxidizing action of nitric acid?*

Pour 1-2 cc. of the acid on a piece of white woolen yarn in a test tube, rinse with water, and then add ammonium hydroxide. This action of nitric acid followed by ammonium hydroxide serves as a **test** for those nitrogenous substances known as **proteins**.

Pour 1-2 cc. of the acid on a small piece of copper in a test tube. This action is characteristic of nitric acid. Repeat this experiment using zinc instead of copper. *Compare the action of nitric acid on metals with that of hydrochloric and sulphuric acids.*

c. **Test for the nitrate ( $\text{NO}_3^-$ ) ion.** This is a very delicate test, and the directions must be followed with care. Pour into a test tube about 3 cc. of ferrous sulphate ( $\text{FeSO}_4$ ) solution. Add to it 2 drops of dilute nitric acid and shake the test tube. Holding the tube in an inclined position, pour 2 cc. of concentrated sulphuric acid slowly down the side of the tube (Fig. 60). The heavy acid will run down the tube and form a separate layer under the mixture of the other two solutions. *Describe the coloration in the ring where the two layers meet.* Repeat this test using a very small amount of sodium nitrate dissolved in 2 cc. of water instead of the dilute nitric acid.

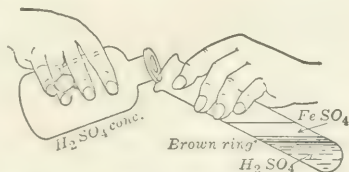


Fig. 60. Testing for nitrate ions with ferrous sulphate solution.

d. **Summarize** the properties of nitric acid.

**Optional work.** Investigate and compare the action of dilute nitric acid (1 to 10) and concentrated nitric acid on copper, magnesium, and tin.

## EXPERIMENT 34

### NITRIC OXIDE

**Apparatus.**—Wide-mouth bottle with 2-hole stopper. Thistle tube. Pneumatic trough. 3 wide-mouth bottles and glass squares. Deflagrating spoon. Large test tube with 2-hole stopper. Dropper. Glass tubing.

**Materials.**—Copper turnings. Concentrated nitric acid. Tapers. Asbestos paper. Red phosphorus. Sodium peroxide.

a. **Preparation.** Place some pieces of copper (about 10 g.) in a generating bottle and fit it with a 2-hole stopper carrying a thistle tube and a delivery tube, as shown in figure 61. Prepare to collect 3 bottles of gas over water. Pour in enough

water to cover the copper and the lower end of the thistle tube and then add about half as much concentrated nitric acid. The action may be a bit slow in starting; if necessary add

from time to time small quantities of the concentrated nitric acid. If the action becomes very vigorous, add cold water.

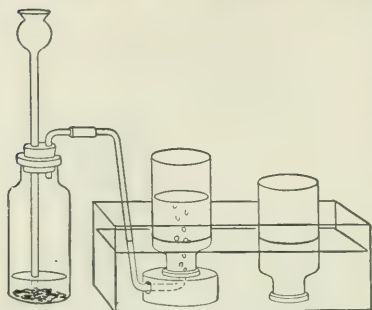


Fig. 61. Making nitric oxide.

After the air in the generator has been swept out, fill two bottles with the gas, nitric oxide (NO), and fill the third bottle half full. Let them stand in the trough for later use.

*Note the color of the gas in the generating bottle just after the action starts and again later. Also observe the color of the liquid left in the generator. This color is characteristic of the cupric ion ( $\text{Cu}^{++}$ ).*

### b. Properties.

Let one bottle of nitric oxide escape into the air. *What evidence of chemical change do you observe?*

Cover a second bottle of the gas with a glass plate and set it upright on the table. Plunge a lighted taper into the gas, withdraw it at once and replace the plate. Line a deflagrating spoon with asbestos paper and put into it a bit

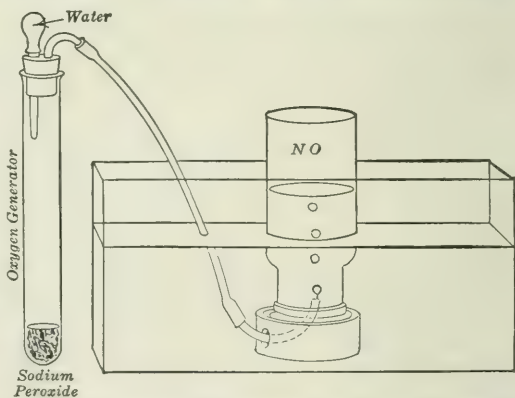


Fig. 62. Generating oxygen and introducing the gas into nitric oxide.

into it a bit

of red phosphorus. Ignite the phosphorus and when it is burning vigorously plunge it into the same bottle. *Does nitric oxide support the combustion of a taper and phosphorus?*

To study the effect of oxygen on nitric oxide, set up a little oxygen generator, as shown in figure 62. Place in the large test tube about 1–2 cc. of sodium peroxide ( $\text{Na}_2\text{O}_2$ ) and fill the dropper with water. Immerse the delivery tube in the trough and let the water drop a little at a time on the peroxide. When the air has been displaced from the test tube, allow the oxygen to ascend slowly into the bottle which is only half full of nitric oxide. *Note the change in color. Does the volume of the gas change? If so, in which direction? Compare the result with that obtained by allowing air to mix with nitric oxide. With what gas in the air does the nitric oxide combine to become brown?* The brown gas is nitrogen dioxide ( $\text{NO}_2$ ).

**c. Summarize** in parallel columns the properties of nitric oxide and nitrogen dioxide.

**Optional work.** Obtain crystals from the liquid left in the generator and find the action of heat on them. Identify the substances produced.

## EXPERIMENT 35

### NITROUS OXIDE

**Apparatus.** — Small flask with 1-hole stopper. Large test tube with 2-hole stopper. Ring stand and clamp. Pneumatic trough. 4 wide-mouth bottles and glass squares. Test tube with 1-hole stopper. Glass tubing.

**Materials.** — Ammonium nitrate. Wooden splint. Copper turnings. Concentrated nitric acid. Anhydrous copper sulphate.

**a. Preparation.** Set up the apparatus as shown in figure 63. Put about 20 g. of ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) into the flask and clamp it with the neck inclined slightly downward. The ammonium nitrate on heating decomposes into water and a

gas, nitrous oxide ( $N_2O$ ). Hence the condensing trap is inserted in the delivery tube. It is better to use warm water in the bottles and trough when collecting this gas.

The heat must be carefully regulated or an explosion may occur. Therefore it is advisable that two pupils work together on this experiment and that one of them give his *entire* attention to heating the flask gently and steadily.

Heat the nitrate gently until it is melted and a good deal of water of crystallization has been driven off. Keep the flame moving since overheating in one spot will cause trouble.

**CAUTION.** If brown fumes appear in the flask during the heating, allow the flask to cool a little.

Let the first jarful of gas escape, as it is mostly air from

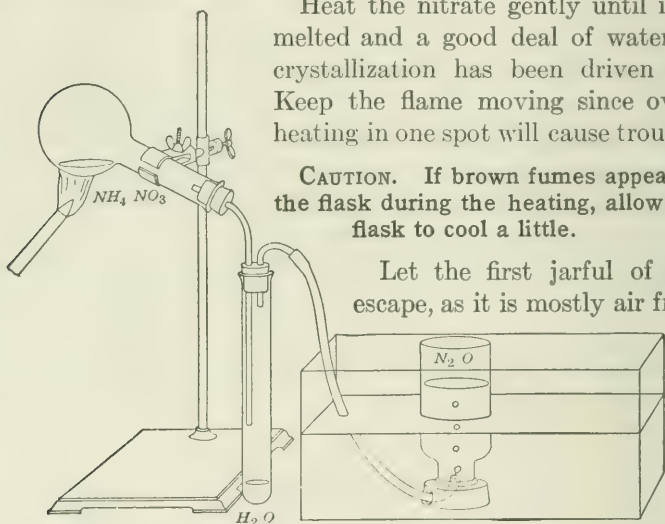
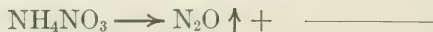


Fig. 63. Generating nitrous oxide.

the flask and test tube. Collect three and a half bottles of the gas and then remove the delivery tube from the water before the heating is discontinued.

Identify the liquid in the large test tube by adding a little anhydrous copper sulphate. Complete the equation:



**b. Properties.** Describe the color and odor of the gas.

Pour a little cold water into a bottle of the gas and cover it

tightly with the palm of the hand. Shake the contents of the bottle vigorously. *What does this experiment show about the solubility of the gas in cold water?*

Plunge a glowing splinter into another bottle of the gas. *What other gas have we already studied which behaves in the same way?*

Prepare some nitric oxide by putting a few pieces of copper turnings into a test tube which is fitted with a stopper and delivery tube. Cover the copper with water, add an equal volume of concentrated nitric acid, and at once insert the stopper. Let the nitric oxide bubble into water until the colored gas in the test-tube generator has disappeared. Then place the end of the delivery tube under the bottle which is half full of nitrous oxide.

*Does the nitrous oxide react on nitric oxide in the same way that we found oxygen did in experiment 34?*

**c. Questions.** (1) *Why is it better to collect nitrous oxide over warm water?* (2) *Why does nitrous oxide support the combustion of a burning splint while nitric oxide does not?* (3) *How would you distinguish between nitric oxide and nitrous oxide? between nitrous oxide and oxygen?*

**Optional work.** Investigate the action of (a) feebly burning sulphur and (b) strongly burning sulphur in nitrous oxide. Explain the difference in action. Is the same compound of sulphur formed when sulphur burns in nitrous oxide that is formed when sulphur burns in air? Give a reason for your answer.

## EXPERIMENT 36

## DESTRUCTIVE DISTILLATION OF COAL

**Apparatus.** — Ring stand with clamp. Ignition tube (hard-glass test tube) with 1-hole stopper. Large test tube with 2-hole stopper. Pneumatic trough. Wide-mouth bottle. Bunsen burner with wing-top. Glass tubing.

**Materials.** — Small pieces of bituminous (soft) coal. Litmus papers. Lead nitrate test papers.

**a. Distillation of coal.** Fill an ignition tube about three-fourths full of small pieces of soft coal and clamp it horizontally

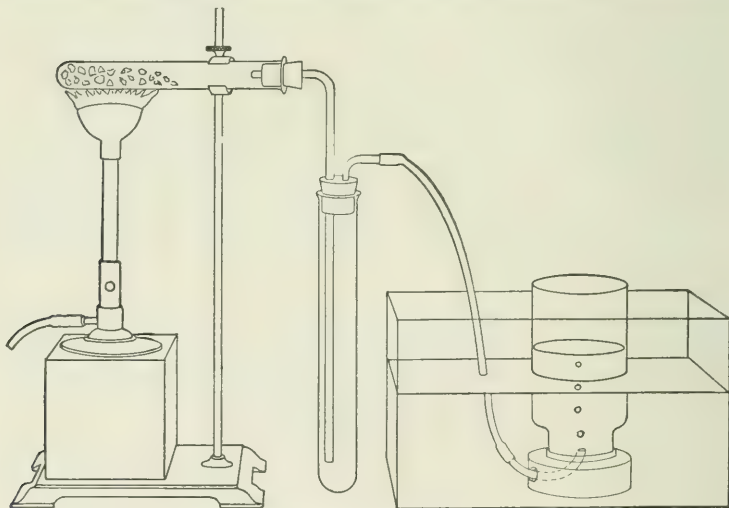


Fig. 64. Destructive distillation of coal.

as shown in figure 64. Have the delivery tube extend nearly to the bottom of the condensing tube (large test tube) and arrange to collect the gaseous products over water. Heat the ignition tube gently at first and then strongly until no more gas is given off. During the heating, test the stream of gas

in the delivery tube for hydrogen sulphide ( $\text{H}_2\text{S}$ ) with a strip of paper moistened in lead nitrate solution, and also for ammonia with moistened red litmus paper. *Results?*

**b. Products.** Examine the gas which has been collected over water. *Does it burn?*

Observe the properties of the material in the condensing tube. *Name it.*

When the ignition tube has become cool enough to handle, shake its contents out on the base of the ring stand. *Compare this residue in color, structure, and weight with the original coal. Name it.*

*Why is this process called destructive distillation?*

*Name three important products of the destructive distillation of coal. Name a by-product.*

**Optional work.** In the same way carry out the destructive distillation of wood, using small splinters or chips. In this process the tarry distillate in the condensing tube is a mixture (called pyroligneous acid) which contains wood alcohol, acetic acid, acetone, tar, and other substances.

## EXPERIMENT 37

## CHARCOAL — PREPARATION AND PROPERTIES

**Apparatus.** — Tripod. Bunsen burner. Sand crucible. Iron crucible and cover. Pipe-stem triangle. Ignition tube with 1-hole stopper. Ring stand and clamp. Test tubes. Mortar and pestle.

**Materials.** — Blocks of wood. Solution of molasses, brown sugar, cochineal, or indigo. Animal charcoal (boneblack). Copper sulphate solution. Hydrogen sulphide solution. Powdered copper oxide. Limewater.

**a. Preparation.** When animal or vegetable matter is heated strongly in the absence of air, the residue is charcoal. We may easily prepare wood charcoal by placing on a thin layer of sand in a sand crucible or on a small iron pan a few chips or small blocks of wood. If we then cover them with sand to keep out the air (Fig. 65) and heat strongly until all smoking ceases, we find, when the crucible is cool, that the residue is charcoal.

**b. Properties.** (1) To show that carbon is a decolorizer, fill a test tube half full of water which has in it enough molasses, brown sugar, cochineal, or indigo to give a decided color. Add about 5 cc. of animal charcoal to the solution, shake thoroughly, and boil for 3–4 minutes. Filter the solution, and, if the filtrate is not clear and transparent, repeat the operation with fresh animal charcoal. The activity of the charcoal is much increased by previous heating in a covered iron crucible. If a solution of sugar or molasses was used, taste the filtrate to determine whether or not the sugar has been removed.

Repeat this experiment with a solution of copper sulphate.

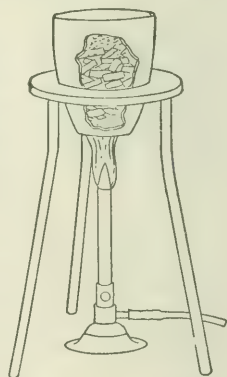


Fig. 65. Preparation of charcoal in a sand crucible.

*Can the color be removed from all liquids by filtering them through animal charcoal (boneblack)?*

(2) To show that carbon is a deodorizer, fill a test tube about one-fourth full of powdered wood charcoal and add about 5 cc. of hydrogen sulphide ( $\text{H}_2\text{S}$ ) solution. Cork the test tube and at short intervals for 15 minutes shake the contents vigorously. Compare the odor with that of the original solution and, if it is

not perceptibly removed, add more charcoal and continue shaking the contents. *What use of this property of charcoal was made in the World War?*

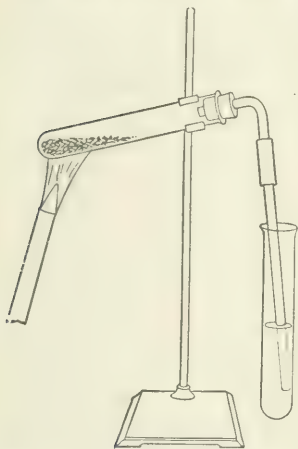


Fig. 66. Reducing copper oxide with carbon.

**c. Uses.** The most important chemical use of charcoal (or coke) is as a reducing agent. Mix intimately about 5 g. of copper oxide ( $\text{CuO}$ ) with 1 g. of powdered wood charcoal in a mortar. By means of a folded strip of paper slide the mixture into a hard-glass test tube (ignition tube) and fit the tube with a 1-hole stopper carrying a delivery tube. Arrange the apparatus as shown in figure 66 so that the gas

evolved when the tube is heated will bubble through a little limewater in a test tube.

Heat the mixture in the ignition tube at first gently to avoid cracking the tube and then as strongly as possible, beginning at the part near the stopper and gradually moving the flame toward the closed end of the tube. The tube must be heated persistently for at least 10 minutes. *What is the first change that takes place in the limewater?*

Remove the delivery tube from the limewater and stop heating. When the tube is cold pour its contents into a mortar and vigorously grind the residue under water. Let a stream

of water wash away the lighter particles. *What substance is carried away by the water? What is left in the mortar? What element is taken from the copper oxide? Describe the process and write the equation of the reaction.*

**d. Results.** *Summarize these properties of charcoal (carbon) which you have observed in this experiment.*

**Optional work.** We may also use carbon to reduce such metallic oxides as tin oxide ( $\text{SnO}_2$ ) and lead oxide ( $\text{PbO}$ ). This same process may be utilized to **detect carbon in a compound**. Find out whether sugar, wax, and naphthalene (moth balls) contain carbon. *How could you utilize this same process to detect hydrogen in the compounds?*

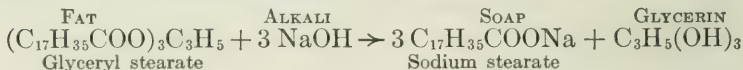
### EXPERIMENT 38

#### THE MAKING OF SOAP—ITS USES

**Apparatus.**—Evaporating dish. Tripod and asbestos gauze. Bunsen burner. Stirring rod. Test tubes and rack.

**Materials.**—Alcohol. Vegetable oil (olive, cottonseed, or cocoanut). Sodium hydroxide solution (20%). Calcium chloride solution. Kerosene.

**a. Preliminary.** Sodium hydroxide ( $\text{NaOH}$ ) is one of the strong bases (an alkali). When fats or vegetable oils are boiled with it, they are saponified, *i.e.* converted into soap. These fats are chiefly glyceryl esters of organic acids. On boiling, the fat is decomposed by the sodium hydroxide into glycerin ( $\text{C}_3\text{H}_5(\text{OH})_3$ ) and the sodium salt of the organic acid, which is soap. The following equation represents such a reaction:



**b. Preparation.** Add 10 cc. of alcohol to about 2 teaspoonfuls of oil (olive, cottonseed, or cocoanut) in an evaporating dish. Heat the dish with a small flame which barely touches the dish. Then add 5 cc. of a 20 per cent solution of sodium hydroxide and continue heating and stirring the contents constantly until the odor of alcohol is no longer perceptible (Fig. 67). The pasty mass which remains in the dish is the soap mixed with glycerin.

**NOTE.** To save time in this experiment alcohol is used as a common solvent for the oil and the alkali. In commercial work it is not used.

**c. Uses.** Wash your hands with a little of the soap you have just made. *Does it lather readily?* When too much alkali has been used, the soap makes the hands rough. An excess of oil makes the soap greasy. *What about the quality of your soap?*



Fig. 67. Making soap.

Make a half test-tubeful of soap solution, add to it half as much calcium chloride ( $\text{CaCl}_2$ ) solution, and shake the liquid. An insoluble calcium soap is formed. This insoluble soap, which is formed whenever soap is used with "hard" water, is useless for cleaning purposes.

Put 1 cc. of kerosene into 10 cc. of water in a test tube and shake the mixture. Note that the oil is broken up into tiny globules which form an **emulsion**. Let the test tube stand for a minute to see whether the emulsion remains; then add about 2-3 cc. of soap solution to the mixture and shake again. *What effect does the soap have on the permanency of the emulsion?* In this same way soap forms an emulsion with the oil which makes dirt cling to the fibers of clothing. This action loosens the dirt so that it can be easily removed by mechanical washers.

**Optional work.** Soap can be made from waste fat. Even though the fat is dirty and rancid, it can be used by melting and straining it through cheesecloth. Weigh the fat and also weigh out about one-third as much commercial lye (Babbitt's). Dissolve the lye in water. Heat the fat in an iron kettle and slowly pour in the lye solution with constant stirring. Some care is required to see that the liquid does not boil over. After it has boiled for 30 minutes, add water to make up for that which has boiled away and then stir in common salt, about twice the weight of the lye used. When cold, remove the layer of soap on top. The soap may be warmed and allowed to cool in small pasteboard boxes so as to form cakes.

## EXPERIMENT 39

## REMOVAL OF SPOTS AND STAINS

**Apparatus.** — Beakers. Test tubes and rack. Bunsen burner.

**Materials.** — Samples of various kinds of cloth (cotton, wool, silk) spotted with grease, sugar, ink, iron rust, etc. Carbon tetrachloride, turpentine, oxalic acid, Javelle water, etc.

**a. Preliminary.** The simplicity of the cleansing process varies with the character of the stain. For example, the removal of a grease spot from cloth is merely a process of solution and absorption; but the removal of a fruit stain or of an ink spot from cloth often involves a careful study of the cloth itself as well as of the various bleaching reagents.

In general there are three methods of treatment: (1) **Warm water** will remove sugar, glue, blood, if not too old; (2) **Gasolene, carbon tetrachloride, or turpentine** will act as a solvent for grease and paint and so can be used to remove such spots; (3) **Bleaching agents**, such as oxalic acid or citric acid, followed by bleaching powder may be used to remove ink spots. Of course it is quite important to remember that gasolene vapor is very inflammable and should never be used in the vicinity of a flame. Some absorbing material must be placed beneath the goods to be cleaned in order that the grease may be washed through by the gasolene. Otherwise, rubbing a little gasolene on a spot merely spreads the grease over a larger area. In using bleaching agents it is necessary to remove with dilute ammonia water the traces of such agents, which might rot the fibers of the cloth.

It is always well to treat the stain on a small sample of the goods before trying to remove the spot or stain from a garment. In general greater care must be exercised with woolen and silk goods than with cotton, and especially with colored goods.

The following table will be useful for reference:

STAIN	REMOVED BY
Sugar, glue, blood	Warm water
Grease	Carbon tetrachloride or gasolene; followed by warm soapsuds and ammonia water
Paint, varnish	Turpentine (except on silk)
Tar, pitch	Soap and oil; followed by turpentine
Inks	Warm oxalic or tartaric acid (20%) or salt and lemon
Iron rust	Warm oxalic or citric acid (10%)
Fruit, tea, and coffee	Hot water. Javelle water *
Acids	Ammonia water to neutralize the acid; then wash with water
Grass	Sponge with alcohol or ammonia water

**b. Practice.** Take samples of 3 or 4 different kinds of cloth and stain them with ink, acid, sirup, and oil. Treat each sample according to the methods just described. Cut each sample down to some convenient but uniform size and mount it with a label in your notebook. Show a sample of the piece of cloth before and after treatment. Also describe briefly just what the treatment was in each case.

**Optional work.** Find out the composition of the two-solution ink eradicant, of the French dry-cleaning fluid, of a reagent for mildew stain, and of a paint and varnish remover. (Consult *Smith & Mess' Laboratory Study of Chemistry*.)

\* Javelle water is made by dissolving a pound of washing soda ( $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$ ) in a quart of boiling water. Add a quarter of a pound of chloride of lime ( $\text{CaOCl}_2$ ) to a quart of hot water. Mix the two solutions and decant the clear solution of sodium hypochlorite ( $\text{NaOCl}$ ) for use.

## EXPERIMENT 40

## TEXTILE FIBERS

**Apparatus.** — Compound microscope. Test tubes and rack. Bunsen burner.

**Materials.** — Samples of cotton, wool, silk, and linen cloth. Samples of cotton and wool mixed goods, cotton and linen mixed goods. Sodium hydroxide solution (20%). Alcoholic solution of fuchsin. Dilute ammonium hydroxide. Nickel hydroxide solution. Dilute hydrochloric acid.

**a. Microscopic study of fibers.** Study carefully the appearance of fibers of cotton, wool, silk, and linen under the microscope. (Compare with figure 170 on page 279 of B. & C.)

**b. Cotton and wool.** Place in separate test tubes a sample (3 cm.  $\times$  3 cm.) of cotton cloth, woolen cloth, and mixed goods. Cover each with sodium hydroxide solution (20%) and boil for 2–3 minutes. Add water when necessary to keep the cloth covered with liquid.

**CAUTION.** Take great care not to let the hot alkali spatter over on to the flesh.

*Which one of these fibers is completely soluble in sodium hydroxide solution?*

**c. Cotton and linen.** Wet a sample of cloth (part linen) with a 1 per cent alcoholic solution of fuchsin, wash well in water, and then moisten with ammonium hydroxide. The linen (flax) fibers in the cloth will be colored red, while the cotton remains uncolored.

**d. Wool and silk.** Put strips of wool and silk in separate test tubes and cover each with a solution of nickel hydroxide.\*

\* **Nickel hydroxide** solution may be prepared by dissolving 5 g. of nickel sulphate in 100 cc. of water and then adding a solution of sodium hydroxide until the nickel hydroxide is completely precipitated. Wash the precipitate well and dissolve it in 25 cc. of concentrated ammonium hydroxide. Then add 25 cc. of distilled water.

After 2 minutes remove the residue and boil for another 2 minutes in a 1% solution of hydrochloric acid. *Which of the samples remains undissolved?*

**e. Silk, wool, and cotton.** *How would you prove the presence of silk, wool, and cotton in a sample?*

**Optional work.** Weigh to a centigram a sample (10 cm.  $\times$  10 cm.) of some mixed goods. First remove the sizing by boiling the cloth for 10 minutes in a 1 per cent solution of hydrochloric acid. Wash and dry the sample and reweigh to determine the per cent of sizing. Then proceed to separate the various fibers as just described and calculate the per cent of each kind of fiber. Mount neatly in your notebook a sample (3 cm.  $\times$  3 cm.) of the cloth analyzed.

*How would you distinguish between real silk and artificial silk?*

## EXPERIMENT 41

### COMPONENTS OF FOODS

**Apparatus.** — Test tubes and rack. Bunsen burner. Mortar and pestle. Large beaker. Funnel. Crystallizing dish. Iron spoon.

**Materials.** — Starch. Iodine solution. Glucose. Fehling's solution (two solutions). Concentrated nitric acid. Concentrated ammonium hydroxide. Egg. Cornmeal. Peanuts. Oatmeal. Ether. Filter papers.

**a. Starch.** Put a pinch of starch into a test tube and fill the tube half full of water. Shake the contents and boil. *Has the starch changed?* Cool the liquid in running water, and, as soon as it is cold, divide it into two parts. To one add a drop of iodine solution (iodine in potassium iodide). The blue color which is produced is the iodine **test for starch**.

**b. Glucose.** Dissolve about 1 cc. of glucose in 10 cc. of water. Add 5 cc. of Fehling's solution and boil for a few minutes. The red precipitate (cuprous oxide,  $\text{Cu}_2\text{O}$ ) is the characteristic **test for glucose**.

**c. Protein.** Place a few bits of the white of a hard-boiled egg in a test tube and add a few drops of concentrated nitric

acid. Wash off the acid with water and pour off the liquid. Then pour a few drops of ammonium hydroxide on the egg. The yellow color is a **test for proteins** (nitrogenous compounds).

**d. Fats and oils.** Half fill a test tube with cornmeal or crushed peanuts and pour in enough ether or benzine (away from flames) to cover the solid to a depth of a centimeter. Stand the test tube in a beaker of hot water and shake the contents of the test tube occasionally. Filter and let the filtrate evaporate spontaneously in a crystallizing dish. The fat or oil extracted remains in the dish.

**e. Mineral matter.** Place about half a teaspoonful of oatmeal in an old iron spoon and heat it (under the hood) until no more fumes are given off, then heat it strongly to burn off the carbon. The white ash contains various mineral compounds.

**f. Examination of a food.** Apply these tests to one or more foods, such as lean meat, bread, beans (dried), and cheese. Record your results in tabular form.

**Optional work.** Water is one of the important components in foods. Devise some method of determining the per cent of water in some article of food, such as an apple or a potato. When your method is approved by the instructor, carry it out and compare your results with those given on page 283 in B. & C.

## EXPERIMENT 42

### FOOD COMPONENTS OF MILK

**Apparatus.** — Evaporating dish. Short stirring rod. Steam bath. Tripod. Bunsen burner. Ring stand. Horn pan balance and weights. Test tubes and rack. Funnel.

**Materials.** — Sample of milk (from home). Acetic acid. Junket tablets. Fehling's solution. Concentrated nitric acid. Concentrated ammonium hydroxide.

**a. Per cent of solids and water.** To determine the percentage of water in milk, we have only to evaporate to dryness a known

weight of milk. During this process a scum forms over the surface of the liquid, which retards evaporation, and so it is well to use a short stirring rod occasionally to break up the scum.

Weigh a small, clean evaporating dish together with a short stirring rod and then pour into it about 20 cc. of milk and weigh again. Evaporate over a steam bath (Fig. 68) to dryness. As soon as the residue is perfectly dry, wipe dry the outside of the dish, cool, and reweigh. From these results compute the percentages of solids and water in the sample of milk taken. (Cow's milk usually contains not less than 12 per cent of solids.)

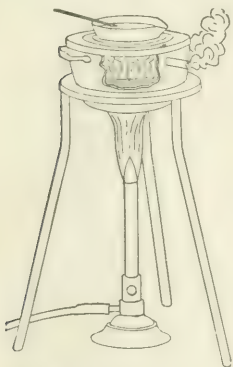


Fig. 68. Evaporating milk over water bath.

**b. Protein.** The principal protein in milk is casein, which is used in the manufacture of cheese. Add 2-3 drops of acetic acid (vinegar may be used) to 10 cc. of milk. When thoroughly mixed and allowed to stand, the casein separates in the form of a curd. When milk sours, the lactic acid formed in the milk causes a natural curdling. Prove that the curd is a protein (Exp. 41).

Another method of separating the casein is by means of rennet. "Junket" tablets contain dried rennet. Dissolve about one-half of such a tablet in water and add the solution to about 100 cc. of warm milk. Again the casein will separate as a curd.

There is another protein in milk which remains in solution when the casein is separated either by an acid or by rennet. This is **albumin**, which is much like the white of egg. Filter off the milk curd (casein) and boil the clear liquid (whey). This coagulates the albumin in the form of white flakes. Prove that the tough scum which forms on the surface of boiled milk is coagulated albumin (a protein).

**c. Sugar of milk (lactose).** The filtrate left after the casein and albumin have been coagulated and removed contains the sugar of milk. By evaporation of this filtrate we obtain a residue. The taste shows that this sugar is only about one-sixth as sweet as cane sugar. Test it with Fehling's solution.

**Optional work.** The most valuable component of milk is doubtless the **butter fat**. The most practical method of determining the per cent of butter fat is the **Babcock test**. In this process a definite quantity (17.6 cc.) of milk is treated with sulphuric acid (17.5 cc.,

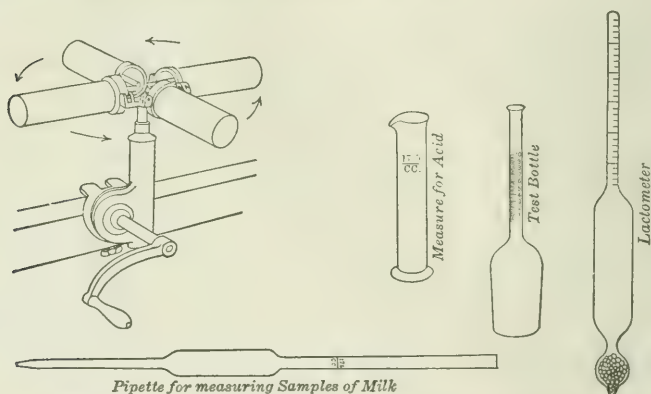


Fig. 69. Milk-testing outfit. Centrifuge. Babcock test bottles and lactometer.

Sp. Gr. 1.83) which dissolves the casein of the milk and thus facilitates the separation of the fat. This mixture is then whirled in a centrifugal machine, and the fat collects on the surface. The necessary apparatus is shown in figure 69. The test bottle is so made that the graduations on the neck indicate the percentage of fat directly. Further directions are furnished with the apparatus.

By using the Babcock test and by determining the specific gravity with a lactometer it is possible to estimate approximately the other solids in milk in addition to the fat. The Babcock formula, which is based on experimental results, is as follows:

$$\text{Solids-not-fat} = \frac{\text{Lactometer}}{4} + 0.2 \text{ Fats}$$

The lactometer commonly used (Quevenne) is so graduated that the scale represents only the second and third decimal places of the specific gravity. For example, if it reads 34 the specific gravity is 1.034; and if the percentage of fat is 3.5 per cent, then the per cent of **solids-not-fats** is  $\frac{34}{4} + 0.2 \times 3.5$  or **9.2**.

## EXPERIMENT 43

### TESTING FOODS FOR ADULTERATIONS

**Apparatus.** — Iron spoon. Bunsen burner. Wooden splint. Small flask. Beaker. Tripod. Asbestos gauze. Test tubes.

**Materials.** — Creamery butter and oleomargarine. 1 pint of sweet milk (from home). Concentrated sulphuric acid with ferric chloride solution (25 cc. acid and 1 cc.  $\text{FeCl}_3$  sol.). Formaldehyde. Cracked ice.

**a. Butter and its substitutes.** Many of the substitutes for butter are wholesome and much cheaper than real butter. But these substitutes should not be sold as butter. The following simple tests will serve to distinguish butter from its substitutes.

**Foam test.** Melt a lump of the so-called butter in a large iron spoon over a small flame. Genuine creamery butter will boil quietly with the production of considerable froth or foam. This is caused by the escape of steam from the butter. Oleomargarine and renovated butter bump and sputter and make more noise, producing little foam. Test a piece of real butter and compare its action with that of a substitute.

**Waterhouse test.** Place about 50 cc. of sweet milk in a small flask and set in hot water to heat. When it is almost boiling, add 4–5 g. (about a teaspoonful) of the sample and stir it with a wooden stick until it melts. Then place the flask in ice water and continue stirring until the fat solidifies. If the sample is **oleomargarine**, the fat can be collected into one lump which can be removed with the stick. **Butter fat** cannot be

so collected, but is more or less emulsified with the milk. If the sample is **renovated butter**, it will tend to collect, when the stirring is stopped, as a film on the top of the milk.

**b. Formaldehyde in milk.** The use of this preservative in milk is especially pernicious because milk is so largely used as food for babies and children. Formaldehyde is one of the most poisonous preservatives found in foods. Milk should be regarded with suspicion if it takes too long to sour.

Bring a pint of milk from home or from a store and have the instructor add 2-3 drops of formaldehyde. Label the bottle and set the milk aside. Record how long it takes the milk to sour.

To test suspected milk for formaldehyde fill a test tube one-third full of the milk. Holding the tube in an inclined position, carefully pour down the side of the tube a little concentrated sulphuric acid to which has been added a drop of ferric chloride solution. The milk will rest upon the surface of the acid and if formaldehyde is present, a violet color forms where the acid and milk come into contact. Apply this test to the milk to which formaldehyde was added.

**Optional work.** Although the subject of food inspection and analysis is one of vital interest to the community, yet it is one which generally requires considerable technical training and experience. Nevertheless there are some simple tests which can be easily applied. The student is referred to the handy little book, by *Lewis B. Allyn*, *Elementary Applied Chemistry*, and to the larger book on the subject, by *A. E. Leach*, *Food Inspection and Analysis*.

## EXPERIMENT 44

## SODIUM CARBONATE AND BICARBONATE

**Apparatus.** — Test tubes. 1-hole stopper to fit test tube. Ring stand and clamp. Bunsen burner. Beakers. Tripod and asbestos gauze. Glass tubing. Horn pan balance and weights.

**Materials.** — Sodium bicarbonate (baking soda). Limewater. Potassium bitartrate (cream of tartar).

**a. Effect of heating the bicarbonate.** Put about 2–3 g. of sodium bicarbonate ( $\text{NaHCO}_3$ ) into a test tube and clamp it so that the mouth is inclined slightly upwards. Arrange the delivery tube so that it leads into a test tube containing limewater ( $\text{Ca}(\text{OH})_2$ ) (Fig. 70). Warm the bicarbonate gently with a small flame, taking care not to heat the test tube hot enough to color the flame yellow.

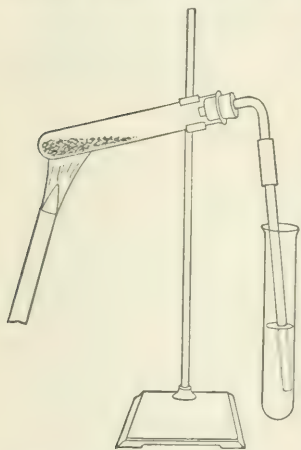


Fig. 70. Heating sodium bicarbonate and passing the gas into limewater.

*What gas is evolved? What collects on the cool part of the test tube? To part of the residue left in the tube, add an acid. What is the result? What is the residue? Write the equation for the action of heat on sodium bicarbonate. Compare the taste of the bicarbonate with that of the residue.*

**b. Baking powder.** Weigh out 2 g. of potassium bitartrate (cream of tartar,  $\text{KHC}_4\text{H}_4\text{O}_6$ ). Calculate the amount of sodium bicarbonate required from the following equation:



Weigh out this amount of sodium bicarbonate and mix the two salts thoroughly on paper.

Put half of this mixture into a beaker half full of *cold* water and the other half into another beaker half full of *hot* water. *What is the gas which escapes? What is the difference between the action of cold and hot water on this mixture? Why is water needed in this chemical reaction?*

**Optional work.** Sodium bicarbonate may be prepared according to the **Solvay process**. Dissolve 10 g. of powdered ammonium carbonate in 100 cc. of cold concentrated ammonium hydroxide. Shake the mixture to secure a solution and then saturate the solution with sodium chloride by shaking the liquid vigorously with finely pulverized salt.

Pour off the clear solution into a bottle and pass slowly a stream of washed carbon dioxide gas through it until a copious precipitate of sodium bicarbonate is produced. Filter, and dry the precipitate by pressing between filter papers.

## EXPERIMENT 45

### ANALYSIS OF BAKING POWDER

**Apparatus.** — Test tubes and rack. Bunsen burner. Evaporating dish. Funnel. Beaker (small). Stirring rod. Ring stand and asbestos gauze.

**Materials.** — Sample of baking powder (2 tablespoonfuls, from home). Iodine solution. Barium chloride solution. Dilute hydrochloric acid. Concentrated sulphuric acid. Concentrated nitric acid. Ammonium molybdate solution. Sodium hydroxide solution. Filter papers.

**a. Preliminary.** Sodium bicarbonate is an essential ingredient of all baking powders. *How could you prove its presence?* The other necessary ingredient is some solid substance which is weakly acidic, such as cream of tartar, calcium acid phosphate, or alum. A small amount of starch or flour is usually added to keep the mixture dry. The different kinds of baking powder vary according to the kind of acid agent present. Some powders have a mixture of two or even of all three of these acid-forming ingredients.

Place about 10 g. of a baking powder in a beaker and add about 50 cc. of water. Stir the mixture well until all the gas is evolved. Then filter and test the filtrate and residue for the various ingredients as follows :

**b. Starch.** *Will the starch be found in the filtrate or in the residue?* Test for starch with one drop of iodine solution as in Exp. 41.

**c. Sulphates.** Ordinary **alum** is a double salt of aluminum sulphate and potassium sulphate ( $\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24 \text{H}_2\text{O}$ ). **Ammonium alum** is also a double salt of ammonium sulphate and aluminum sulphate ( $(\text{NH}_4)_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24 \text{H}_2\text{O}$ ). Thus it will be seen that if a baking powder contains alum, the filtrate will contain sulphates. Test the filtrate with barium chloride solution and dilute hydrochloric acid as in Exp. 31.

**d. Tartrates.** Pour about 5 cc. of the filtrate from (a) into an evaporating dish and add 5 drops of concentrated sulphuric acid. Evaporate to dryness over a small flame. Charring and the odor of burnt sugar indicate the presence of a tartrate.

**e. Phosphates.** If the baking powder contains calcium acid phosphate, it will be found in the filtrate from (a). To test for a phosphate, take a few cubic centimeters of the filtrate and acidify with a few drops of nitric acid. Warm the mixture and add a few drops to 5 cc. of ammonium molybdate solution ( $(\text{NH}_4)_2\text{MoO}_4$ ). Set it aside. A bright yellow precipitate indicates a phosphate.

**f. Ammonium salts.** If the baking powder contains ammonium alum, we can detect it by pouring about 5 cc. of the filtrate into a test tube and adding the same volume of sodium hydroxide solution. If heated to boiling, ammonia gas is evolved.

**g. Calcium and aluminum.** Since a baking powder containing sulphates always contains aluminum and one containing phosphates always contains calcium, we need not make special tests for these metals here.

In making the report of your chemical analysis, state *where*

*you obtained it, the name of the manufacturer, exact name of brand, what you tested for and how, and what you found present.*

**Optional work.** Compute the cost of making tartrate baking powder according to the following formula: 1 lb. sodium bicarbonate, 2 lb. cream of tartar, 1 lb. cornstarch. Use the prices given in a trade catalogue. Would it be worth while to prepare baking powder for home use? Why should it be kept in air-tight containers?

Look up the formulas of some other baking powders and compute the cost of ingredients.

## EXPERIMENT 46

### HYDROLYSIS OF SALTS

**Apparatus.** — Test tubes and rack. Stirring rod.

**Materials.** — Litmus papers (red and blue). Copper sulphate, aluminum sulphate, ferric chloride, sodium chloride, potassium nitrate, sodium carbonate, and potassium carbonate.

**a. Preliminary.** Not all salt solutions react neutral when tested with litmus paper. This is due to the fact that water is itself very slightly dissociated into  $H^+$  and  $OH^-$ . The action of the ions of water on a dissolved salt is called hydrolysis. When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak base, an equivalent amount of hydrogen ions ( $H^+$ ) is set free and the solution gives an **acid** reaction with litmus. When the ions of the salt unite with the ions of water to form the undissociated molecules of a weak acid, an equivalent amount of hydroxyl ions ( $OH^-$ ) is set free and the solution gives a **basic** reaction with litmus.

NOTE. Remember that the reddening of blue litmus shows  $H^+$  and the turning blue of red litmus shows  $OH^-$ .

**b. Testing salt solutions with litmus.** Dissolve about 0.5 g. of each of the following substances in 10 cc. of distilled water in separate test tubes: copper sulphate ( $CuSO_4$ ), sodium chloride

(NaCl), sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), aluminum sulphate ( $\text{Al}_2(\text{SO}_4)_3$ ), ferric chloride ( $\text{FeCl}_3$ ), potassium nitrate ( $\text{KNO}_3$ ), and potassium carbonate ( $\text{K}_2\text{CO}_3$ ).

Test each solution with litmus paper by putting a small drop of the liquid on the paper with a clean glass rod. *Divide these solutions into three classes:* (1) those which give an *acid* reaction, (2) those that give a *basic* reaction, and (3) those that are *neutral*. Take one case from each of these three classes and *explain in terms of the ionic theory the reaction of the solution toward litmus. Write equations to illustrate hydrolysis in classes 1 and 2.*

## EXPERIMENT 47

### FLAME TESTS

**Apparatus.**—Bunsen burner. Platinum wire (iron or nichrome) mounted in a glass tube. Test tubes. Cobalt glass (2 or 3 squares).

**Materials.**—Concentrated hydrochloric acid. Chlorides of sodium, potassium, lithium, calcium, strontium, and barium. Unknowns.

**a. Preliminary.** There are certain metals which are easily detected, in whatever salt they may occur, because of a characteristic color which each imparts to the Bunsen flame. This furnishes a convenient and often a very delicate qualitative test for these metals. We shall study compounds of **sodium, potassium, lithium, calcium, strontium, and barium**. We may use a piece of platinum wire which has a little hook bent at one end and is melted into a short piece of glass tube at the other end. (Nichrome wire may be substituted here, and one end may be twisted around a match stick to serve as a handle.)

Hold the end of the platinum wire in the blue Bunsen flame. If the flame is colored by it, dip the wire in a little concentrated hydrochloric acid in a test tube and again hold it in the flame. Repeat this several times until the wire ceases to impart any color to the flame.

**b. Sodium.** Heat the end of the cleaned wire and quickly touch a tiny particle of sodium chloride. Hold the wire with the adhering particle in the lower part of the outer blue layer of the flame (Fig. 71) and *note the color of flame produced*. Examine the sodium flame through two thicknesses of blue (cobalt) glass. *What tint of light is absorbed by the blue glass and hence does not reach the eye?* Hold the wire in the flame until the color has nearly disappeared, then clean it as before by dipping in hydrochloric acid.

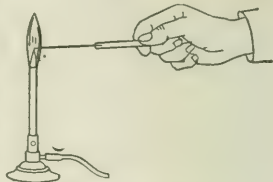


Fig. 71. Flame test for sodium, etc.

**c. Potassium.** When the wire is quite clean, heat it and touch a bit of potassium chloride. *Record the color imparted to the flame*. Hold two thicknesses of blue glass between the flame and the eye and observe whether the color is absorbed.

Mix together intimately a little sodium chloride and potassium chloride and plunge the hot wire into the mixture. *Record the color imparted to the flame. Why do you not see both of the characteristic colors?* Now examine the flame of the mixture through blue glass. *Which metal can you recognize in this way?*

**d. Lithium, calcium, strontium, and barium.** When the wire is thoroughly clean and imparts no color to the flame, plunge it into a little lithium chloride and hold it in the flame. *Note the characteristic color.*

Repeat this experiment with the chlorides of the next three metals (Ca, Sr, and Ba), and tabulate your results:

SUBSTANCE	FORMULA	COLOR OF FLAME	REMARKS

**e. Test for unknown.** Obtain from the instructor a numbered envelope containing an "unknown." This may contain

one or two salts of these six metals. *Record the number of your unknown and identify it.*

**Optional work.** If a spectroscope (Fig. 72) is available, study the spectra of these metals and compare your results with those shown on the Spectrum Chart (facing page 304, B. & C.).

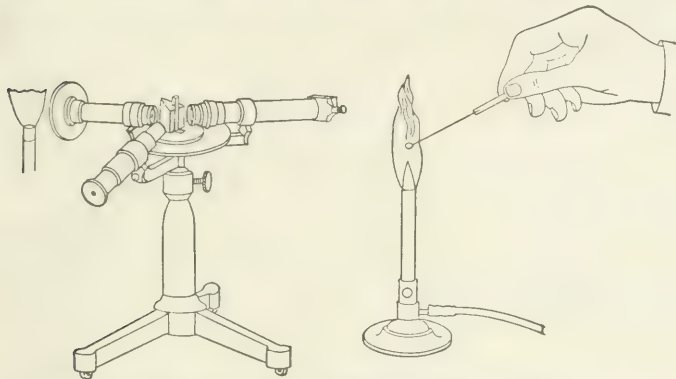


Fig. 72. Spectroscope used in spectrum analysis.

## EXPERIMENT 48

### CHLORINE — PREPARATION AND PROPERTIES

**NOTE.** If the laboratory is not provided with suitable hoods, this experiment should be done by the instructor.

**Apparatus.** — Flask with 2-hole stopper. Ring stand with large ring and clamp. Thistle tube. Bunsen burner. Pan or water bath. 4 wide-mouth bottles and glass plates. Pneumatic trough. Mortar and pestle. One hydrogen generator for class. Glass tubing.

**Materials.** — Manganese dioxide. Concentrated hydrochloric acid. Powdered antimony. Taper.

**CAUTION.** Chlorine is a poisonous gas. Avoid inhaling much chlorine. Breathing ammonia gives relief. This experiment should be done under a hood or in a place where there is a good draft.

**a. Preparation.** Set up the apparatus as shown in figure 73. Place about 10 g. of granular manganese dioxide in the generating flask and pour in about 25 cc. of concentrated hydrochloric acid. Rotate the flask so as to mix the contents thoroughly.

Heat the water under the flask to boiling. Fill 4 wide-mouthed bottles by the displacement of the air, holding a piece of white paper behind the bottles to see when they are full. *Note the color of the gas.* Cover the bottles with glass plates and set them aside.

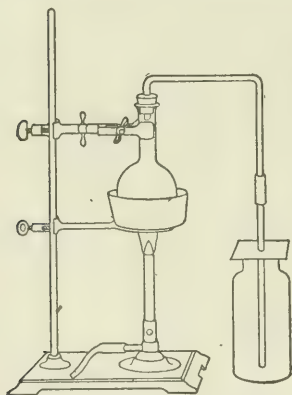


Fig. 73. Preparation of chlorine.

**b. Properties. Solubility in water.** Slide one of the glass plates aside and fill one bottle of chlorine about one-third full of cold water. Cover it with the palm of the hand and shake the contents vigorously. Lower the bottle into a trough of water and remove the hand. *Compare the solubility of chlorine with that of hydrogen chloride (Exp. 16).*

**Action with metals.** Grind up in a mortar a fragment of antimony and sprinkle some of the powdered antimony into a bottle of chlorine. *Name the product and write the equation. Is oxygen always necessary for combustion?*

**Action with hydrogen.** Introduce a jet of burning hydrogen into a bottle of chlorine. Blow gently across the mouth of the bottle and note the change of color. *Name the product and write the equation for the burning.*

**Action with hydrocarbons.** Lower a lighted taper or candle into a bottle of the gas by means of a wire handle. Blow gently across the mouth of the bottle. *Name two products of this action.*

**c. Summarize the properties of chlorine which you have observed in this experiment.**

**Optional work.** Since chlorine is prepared by the oxidation of the hydrogen in hydrochloric acid, we can use various oxidizing agents. Place a few crystals of potassium permanganate in a beaker and add 2-3 cc. of concentrated hydrochloric acid. Cover the beaker with a glass plate. After a few minutes examine the beaker and identify the gas.

Repeat the experiment using in turn powdered potassium chlorate, potassium dichromate, and lead dioxide, as the oxidizing agent.

## EXPERIMENT 49

### BROMINE — PREPARATION AND PROPERTIES

**Apparatus.** — Ring stand and clamp. Test tubes and rack. 1-hole stopper to fit test tube. Wide-mouth bottle. Bunsen burner. Glass tubing.

**Materials.** — Potassium (or sodium) bromide. Manganese dioxide. Concentrated sulphuric acid. Carbon disulphide. Chlorine water (saturate water with the gas).

**a. Preparation.** Fit a cork stopper to a test tube and bore a hole through it. Insert a delivery tube and set up the apparatus



Fig. 74. Preparation of bromine.

as shown in figure 74. Mix on a piece of paper about 1 cc. of potassium bromide (or sodium bromide) crystals with twice this bulk of manganese dioxide and put the mixture into the test tube. Add about 2 cc. of concentrated sulphuric acid to 1 cc. of water in a test tube and cool it. Then pour the acid on the mixture of potassium bromide and manganese dioxide. Insert the stopper carrying a delivery tube which extends to the bottom of an empty test tube standing in cold water.

## 100 BROMINE — PREPARATION AND PROPERTIES

Heat the mixture very gently but not enough to boil the black liquid through the delivery tube. Stop heating when all the red vapor is driven over.

**b. Properties.** *Note the color and odor (cautiously) of bromine vapor and the color of the liquid bromine.*

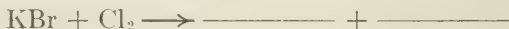
Fill the test tube containing the bromine about two-thirds full of water. Set the mixture aside.

*Is bromine heavier or lighter than water? Is it soluble in water?*

Pour about 2–3 cc. of the “bromine water” just prepared into a test tube and add not more than 1 cc. of carbon disulphide. Cover the tube with the thumb and shake the contents vigorously. Most of the carbon disulphide settles to the bottom. *Compare the colors of the disulphide and water and so the relative solubility of bromine in these two liquids.*

**c. Test for a bromide.** Dissolve a small crystal of potassium (or sodium) bromide in about 5 cc. of water. *Note the color of the solution.* Add 2 cc. of carbon disulphide and shake the mixture. *Why is the carbon disulphide not colored by the bromine?*

Now add a few drops of freshly prepared chlorine water and shake the contents vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete the equation*



**d. Summarize** the properties of bromine: *color, odor, density of vapor (compared with air), density of liquid (compared with water), relative solubility in water and carbon disulphide, and activity compared with chlorine.*

## EXPERIMENT 50

### IODINE — PREPARATION AND PROPERTIES

**Apparatus.** — Test tubes and rack. Bunsen burner. Evaporating dish. Tripod. Crystallizing dish. Asbestos gauze. Mortar and pestle.

**Materials.** — Potassium iodide. Manganese dioxide. Concentrated sulphuric acid. Alcohol. Potassium iodide solution. Carbon disulphide. Chlorine water. Bromine water. Sodium thiosulphate solution.

**a. Preparation.** Pour into an evaporating dish about 3 cc. of water and add about twice that volume of concentrated sulphuric acid. Pulverize several crystals (about 2 g.) of potassium iodide with twice as much manganese dioxide and add the mixture to the acid. Set the evaporating dish on the asbestos gauze supported on the tripod and place a crystallizing dish half full of cold water upon it in such a way that the iodine vapor may be condensed on the bottom of the cool dish (Fig. 75).

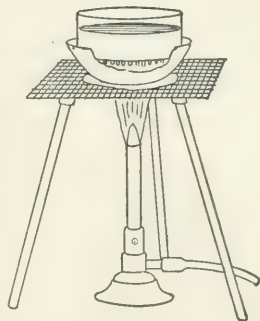


Fig. 75. Preparation of iodine.

Warm the evaporating dish with a very small flame. If the iodine vapor (violet) escapes, decrease the heat and refill the crystallizing dish with cold water. The iodine vapor condenses as a solid (*i.e.* sublimes) on the bottom of the cold dish. Scrape off the iodine crystals on a paper.

**b. Properties.** — **Solvents.** Try to dissolve a crystal of iodine in water by shaking it with a few cubic centimeters of water. *Is the iodine quite insoluble in water?*

To the test tube containing water and iodine, add an equal volume of carbon disulphide and shake the mixture. *Compare the solubility of iodine in water and in carbon disulphide.*

Try the solubility of iodine in ethyl alcohol; also in solutions of potassium iodide and sodium thiosulphate ("hypo"). Tabulate the solubilities of iodine in these various solvents and the colors of the solutions as follows:

SOLVENT	COLOR OF SOLUTION	SOLUBILITY
Water Carbon disulphide, etc.		Very great, moderate, or slight

**c. Replacement of iodine.** Dissolve a small crystal of potassium iodide in about 5 cc. of water and then add a few cubic centimeters of carbon disulphide. Shake the mixture. *Why is the liquid not colored with the iodine?*

Add a few drops of chlorine water and shake the liquids vigorously. Allow the mixture to settle. *What substance has been set free by the chlorine? Complete the equation.*



To another portion of potassium iodide solution, add bromine water and carbon disulphide. After shaking the mixture let it settle. *What substance has been set free by the bromine? Complete the equation.*



*Assuming that the more active element replaces the less active element, which of these halogens is the most active and which is the least active?*

**d. Summarize** the properties of iodine: *color of solid, color of vapor, density of vapor (compared with air), density of solid (compared with water), and relative solubilities in the various solvents used.*

## EXPERIMENT 51

## THE HYDROGEN HALIDES

**Apparatus.** — Test tubes and rack. Bunsen burner.

**Materials.** — Sodium chloride. Sodium or potassium bromide. Sodium or potassium iodide. Concentrated sulphuric acid. Blue litmus paper. Concentrated ammonium hydroxide. Filter papers.

**a. Hydrogen chloride.** Put about 1 g. of common salt (sodium chloride,  $\text{NaCl}$ ) in a test tube and add a few drops of concentrated sulphuric acid. If no action is visible, add a few drops more of sulphuric acid and warm the tube gently. *What happens?*

Blow across the mouth of the tube. *What is the result?* Hold a strip of moistened blue litmus paper near the mouth of the tube. *Result?* Hold a piece of filter paper which has been moistened in ammonia water near the tube. *Explain the formation of a white cloud.*

*Write the equation for the preparation of hydrogen chloride.*

**b. Hydrogen bromide.** Repeat in every detail the experiment (a) using potassium bromide. *Answer the same questions.* Remember that all the hydrogen halides are colorless. *How do you account for the colored gas in the test tube? What element gives this color? What does it indicate as to the stability of hydrogen bromide? What gas is formed besides hydrogen bromide? (Smell the gas very cautiously.)*

**c. Hydrogen iodide.** Again repeat the experiment as in part (a) using potassium iodide. *Answer the same questions.* The amount of the fuming when you blow across the mouth of the test tube indicates the quantity of hydrogen iodide coming from the tube.

*What is the colored vapor?*

*What gas, which you can recognize by its odor, is formed besides hydrogen iodide?*

**d. Comparison.** *What does this experiment show with regard to the relative stability of hydrogen chloride, hydrogen bromide, and hydrogen iodide?*

*Why are hydrobromic and hydriodic acids not usually prepared in this way?*

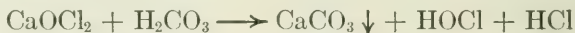
## EXPERIMENT 52

### HYPOCHLOROUS ACID — BLEACHING

**Apparatus.** — Test tubes and rack. Carbon dioxide generator (Exp. 12). Funnel.

**Materials.** — Bleaching powder (chloride of lime). Filter papers. Marble chips. Concentrated hydrochloric acid. Colored calico. Litmus paper.

**a. Preparation.** Make a saturated solution of bleaching powder (chloride of lime) by adding about 2 cc. of the powder to 20 cc. of water in a test tube and by shaking occasionally. Filter off the clear solution and pass carbon dioxide through the liquid for at least 5 minutes. The equation for this reaction is as follows:



By filtering off the precipitate, calcium carbonate, we obtain a clear solution containing the hypochlorous acid.

**b. Use in bleaching.** Place in the solution of the hypochlorous acid, just prepared, small strips of (1) litmus paper, (2) paper with printing on it, (3) paper on which there is writing in ink, (4) paper with pencil marks, and (5) colored calico. *Record the effect on each. Which of these materials is colored with organic compounds and which with free carbon? Does hypochlorous acid oxidize free carbon? What does it oxidize?*

**Optional work.** Unbleached cotton cloth may be bleached with chloride of lime as follows:— (1) First clean the cloth of grease by boiling the cloth in a weak solution of washing soda (5%  $\text{Na}_2\text{CO}_3$  solution). (2) Then wash and place the cloth in a bleach

bath (10%  $\text{CaOCl}_2$  solution). (3) After 5 minutes partially wring and place the cloth in a very dilute solution of sulphuric acid (5 cc. conc.  $\text{H}_2\text{SO}_4$  in 100 cc. water). (4) Again place the cloth in the bleach solution and thus alternate until the color is removed. (5) To remove any traces of the hypochlorous acid left in the cloth, wash it several times in a solution of sodium sulphite (5%  $\text{Na}_2\text{SO}_3$  sol.). Mount in your notebook a sample of the original cloth and another as bleached.

## EXPERIMENT 53

### HYDROGEN PEROXIDE

**Apparatus.** — Small flask. Stirring rod. Glass jar. Funnel. Test tubes and rack.

**Materials.** — Sodium peroxide. Cracked ice. Litmus papers. Dilute hydrochloric acid. Powdered manganese dioxide. Wooden splint. Lead nitrate solution. Ammonium sulphide solution. Potassium dichromate solution. Dilute sulphuric acid. Ether.

**a. Preparation.** Dissolve in a flask about 1 cc. of sodium peroxide in 100 cc. of ice-cold water. Add the sodium peroxide to the water in very small quantities with constant stirring and cooling in running water. Test a drop of the solution with litmus paper. Now add a few drops of hydrochloric acid and shake and cool the flask. Continue this process until the solution is slightly acid. *What are the products of this reaction? Write the equation.*

**b. Properties. Stability.** Take half a test-tubeful of the solution and have ready on a piece of paper about 1 cc. of finely powdered manganese dioxide. Light a wooden splint and then put the manganese dioxide into the solution. Test the gas in the test tube for oxygen with the glowing splint. The manganese dioxide is unchanged and serves as a catalyst. *Is hydrogen peroxide stable or not?*

**Oxidizing agent.** Precipitate some lead sulphide by adding a few drops of ammonium sulphide solution ( $(\text{NH}_4)_2\text{S}$ ) to a few cubic centimeters of lead nitrate solution ( $\text{Pb}(\text{NO}_3)_2$ ). Collect

the precipitate on a filter paper and wash with water 2-3 times. Then pour over the precipitate some of the hydrogen peroxide solution. *What lead compound is formed? Write the equation.*

**c. Test for hydrogen peroxide.** Nearly fill a large test tube with water and add just enough potassium dichromate solution to tinge it a light yellow. Now add a couple of drops of dilute sulphuric acid to set free dichromic acid. Take a portion of hydrogen peroxide solution in a test tube and add 2-3 cc. of ether.

**CAUTION.** Have no flames near when using ether.

Close the tube with the thumb and shake the liquids. Add one drop of dichromic acid solution and shake the liquids again. If in doubt about the result, add another drop of the dichromic acid solution. This is one of the most characteristic and delicate tests for hydrogen peroxide.

**Optional work.** Repeat this experiment using instead of sodium peroxide **lead dioxide**, **barium dioxide**, and powdered **manganese dioxide**. *Is hydrogen peroxide formed in each case? What are the differences in behavior and constitution between a true peroxide and other oxides?*

## EXPERIMENT 54

## TREATMENT OF HARD WATER

**Apparatus.** — Carbon dioxide generator (Exp. 12). Large test tube.

Test tubes and rack. 2 small beakers. Funnel. Bunsen burner.

**Materials.** — Limewater. Marble chips. Concentrated hydrochloric acid. Calcium sulphate solution. Magnesium sulphate solution. Sodium carbonate solution. Filter papers. Concentrated ammonium hydroxide. Ammonium oxalate solution. Ammonium chloride solution. Solution of di-sodium phosphate.

**a. Temporary hardness.** Dilute about 10 cc. of saturated limewater by adding an equal volume of water. Pass a steady stream of carbon dioxide through the half-saturated limewater until the precipitate ( $\text{CaCO}_3$ ) is formed and redissolved. The clear liquid contains calcium bicarbonate ( $\text{CaH}_2(\text{CO}_3)_2$ ) and is called water of temporary hardness. *Write two equations to represent these changes.*

**b. Removing temporary hardness.** To about 5 cc. of the hard water just prepared, add about 3 cc. of saturated limewater and shake the mixture. *What is the precipitate? Write the equation.*

Boil for several minutes about 5 cc. of the hard water prepared in (a). The acid salt (bicarbonate) is changed back into the normal salt with the loss of carbonic acid. Set the tube with contents aside to settle. *Write the equation. Compare the amounts of precipitated calcium carbonate formed by boiling and adding limewater. Explain.*

**c. Permanent hardness.** Pour 10 cc. of a solution of calcium sulphate\* into a small beaker and the same volume of magnesium sulphate solution into another beaker. Add to each 5 cc. of sodium carbonate solution. (Compare solubility of calcium

\* The calcium sulphate solution should be prepared some time beforehand. Shake some plaster of Paris with water, let it stand for some time and then filter.

carbonate and sulphate. Table in Appendix.) Filter each solution. *What is the precipitate in each case? Write the equations.*

Add to the filtrate from the calcium sulphate solution 5 cc. of concentrated ammonium hydroxide and 5 cc. of a solution of ammonium oxalate. If the calcium ion ( $\text{Ca}^{++}$ ) is present, a white precipitate of calcium oxalate will be formed.

Add to the filtrate from the magnesium sulphate solution 5 cc. of a solution of ammonium chloride, 5 cc. of concentrated ammonium hydroxide, and 5 cc. of a solution of di-sodium phosphate. If the magnesium ion ( $\text{Mg}^{++}$ ) is present, a white precipitate of magnesium ammonium phosphate will be formed.

*What does this experiment show in regard to "softening" permanent hard waters with sodium carbonate?*

**Optional work.** Test the temporary and permanent hard waters with soap solution to determine the readiness with which they form permanent soap suds. Also test with the soap solution the hard waters, after they have been softened. *What is the objection to calcium soap? Why are magnesium salts particularly bad in waters to be used in boilers?*

## APPENDIX

### PRESSURE OF WATER VAPOR, OR AQUEOUS TENSION

(In millimeters of mercury)

TEMPERATURE	PRESSURE	TEMPERATURE	PRESSURE	TEMPERATURE	PRESSURE
0° C.	4.6 mm.	11° C.	9.8 mm.	22° C.	19.8 mm.
1°	4.9	12°	10.5	23°	21.0
2°	5.3	13°	11.2	24°	22.3
3°	5.7	14°	12.0	25°	23.7
4°	6.1	15°	12.8	26°	25.1
5°	6.5	16°	13.6	27°	26.7
6°	7.0	17°	14.5	28°	28.3
7°	7.5	18°	15.5	29°	29.9
8°	8.0	19°	16.5	30°	31.7
9°	8.6	20°	17.5	50°	92.3
10°	9.2	21°	18.6	100°	760.0

### DENSITIES OF IMPORTANT GASES

*Approximate weight of 1 liter under standard conditions*

Acetylene ( $C_2H_2$ )	1.16 grams
Air	1.29 "
Ammonia ( $NH_3$ )	0.77 "
Carbon dioxide ( $CO_2$ )	1.98 "
Carbon monoxide (CO)	1.25 "
Chlorine ( $Cl_2$ )	3.17 "
Helium (He)	0.18 "
Hydrogen ( $H_2$ )	0.09 "
Hydrogen chloride (HCl)	1.64 "
Hydrogen sulphide ( $H_2S$ )	1.54 "
Methane ( $CH_4$ )	0.72 "
Nitric oxide (NO)	1.34 "
Nitrogen ( $N_2$ )	1.25 "
Nitrous oxide ( $N_2O$ )	1.98 "
Oxygen ( $O_2$ )	1.43 "
Sulphur dioxide ( $SO_2$ )	2.93 "

## THE METRIC SYSTEM

During the last hundred years most of the civilized nations of the world, except Great Britain and the United States, have adopted the **metric system**, and even in these countries most of the scientific work is done in the metric system. In this system of weights and measures the relation of the units is expressed in multiples of **ten**. Fortunately our system of money is made on the decimal plan, that is, in multiples of ten.

**Some important units of length.** The unit is the **meter** which is equal to 39.37 inches.

$$1 \text{ meter (m.)} = 100 \text{ centimeters (cm.)}$$

$$1 \text{ centimeter} = 10 \text{ millimeters (mm.)}$$

$$1 \text{ kilometer (km.)} = 1000 \text{ meters}$$

$$2.54 \text{ centimeters} = 1 \text{ inch}$$

The chemist needs to become familiar with the centimeter and millimeter. Thus the barometer may read 760 mm. or 76 cm. or about 30 inches.

**Units of volume or capacity.** The unit of volume, that is most extensively used in the laboratory, is the volume of a cube of which the edge is 1 centimeter. This is called a **cubic centimeter** (cc.).

$$1 \text{ liter (l.)} = 1000 \text{ cubic centimeters (cc.)}$$

$$1 \text{ liter} = 1.06 \text{ quarts}$$

Thus a 500 cc. flask holds half a liter or about a pint (16 ounces of water).

**Units of Weight.** The unit of weight is the **gram** which is approximately the weight of one cubic centimeter of water.

$$1 \text{ gram (g.)} = 1000 \text{ milligrams (mg.)}$$

$$1 \text{ gram} = 100 \text{ centigrams (cg.)}$$

$$1 \text{ kilogram (kg.)} = 1000 \text{ grams}$$

$$1 \text{ kilogram} = 2.2 \text{ pounds}$$

$$1 \text{ ounce (avoirdupois)} = 28.35 \text{ grams}$$

The chemist usually expresses weights in grams and decimal fractions thereof, thus a crucible may weigh 12.53 g. Every student in chemistry needs to become very familiar with **centimeters**, **cubic centimeters**, and **grams**.

## TABLE OF SOLUBILITIES

S, soluble in water. P, slightly soluble in water.

I, insoluble in water. Ia, insoluble in water and dilute acids.

	ACETATE	BROMIDE	CARBONATE	CHLORIDE	HYDROXIDE	IODIDE	NITRATE	OXIDE	PHOSPHATE	SULPHATE	SULPHIDE
Aluminum . . .	S	S	—	S	I	S	S	I	I	S	—
Ammonium . . .	S	S	S	S	S	S	S	—	S	S	S
Barium . . .	S	S	I	S	P	S	S	—	I	Ia	S
Calcium . . .	S	S	I	S	P	S	S	—	I	P	P
Copper . . .	S	S	I	S	I	S	S	I	I	S	Ia
Ferrous(Fe <sup>++</sup> )	S	S	I	S	I	S	S	I	I	S	I
Ferric (Fe <sup>+++</sup> )	—	S	—	S	I	—	S	I	I	S	—
Lead . . .	S	P	I	P	I	I	S	I	I	Ia	Ia
Magnesium . .	S	S	I	S	I	S	S	I	I	S	I
Manganese . .	S	S	I	S	I	S	S	I	P	S	I
Mercurous (Hg <sup>+</sup> ) . . .	P	I	I	I	—	I	S	I	I	P	Ia
Mercuric (Hg <sup>++</sup> ) . . .	S	S	I	S	I	I	S	I	I	S	Ia
Nickel . . .	S	S	I	S	I	S	S	I	I	S	I
Potassium . .	S	S	S	S	S	S	S	S	S	S	S
Silver . . .	S	Ia	I	Ia	—	Ia	S	I	I	P	Ia
Sodium . . .	S	S	S	S	S	S	S	S	S	S	S
Stannous (Sn <sup>++</sup> ) . . .	S	S	—	S	I	S	—	I	I	S	Ia
Stannic (Sn <sup>++++</sup> ) . .	S	S	—	S	I	S	—	I	I	—	Ia
Zinc . . .	S	S	I	S	I	S	S	I	I	S	I

## GENERAL RULES FOR SOLUBILITY

(The exceptions to these rules are few and unimportant.)

1. All sodium, potassium, and ammonium compounds are soluble in water.
2. All nitrates, chlorates, and acetates are soluble in water.
3. All chlorides are soluble, except those of silver, mercury (mercurous), and lead (lead slightly soluble).
4. All sulphates are soluble, except those of barium, lead, and calcium (calcium slightly soluble). The silver and the mercurous sulphates are only moderately soluble.
5. All carbonates are insoluble, except those of sodium, potassium, and ammonium.
6. All oxides and hydroxides are insoluble, except those of ammonium, sodium, potassium, and barium (calcium hydroxide slightly soluble).

## EXPERIMENTS AND CORRESPONDING CHAPTERS

The Arabic numerals refer to *Black's* Laboratory Experiments, and the Roman numerals to *Black and Conant's* Practical Chemistry.

EXPERIMENTS	CHAPTERS	EXPERIMENTS	CHAPTERS
1, 2	I	28, 29	XVII
3, 4	II	30, 31	XVIII
5, 6	III	32	XIX
7, 8, 9	IV	33, 34, 35	XX
10	V	36, 37	XXII
11	VI	38, 39	XXIII
12, 13	VII	40, 41, 42, 43	XXIV
14, 15	VIII	44, 45, 46, 47	XXV
16, 17, 18	XII	48, 49, 50, 51	XXVI
19, 20	XIII	52, 53	XXVII
21, 22, 23, 24, 25	XV	54	XXIX
26, 27	XVI		

## TABLE OF THE MORE IMPORTANT ELEMENTS

with their symbols, approximate atomic weights, and valences

Element	Sym- bol	Approx. At. Wt.	Valence	Element	Sym- bol	Approx. At. Wt.	Valence
Aluminum .	Al	27	III	Manganese	Mn	55	II IV
Barium . .	Ba	137	II	Mercury .	Hg	200	I II
Bromine .	Br	80	I	Nickel . .	Ni	58.7	II
Calcium .	Ca	40	II	Nitrogen .	N	14	III V
Carbon . .	C	12	IV	Oxygen .	O	16	II
Chlorine .	Cl	35.5	I	Phosphorus	P	31	III V
Chromium .	Cr	52	II III IV	Platinum .	Pt	195	IV
Copper . .	Cu	63.6	I II	Potassium	K	39	I
Fluorine .	F	19	I	Radium .	Ra	226	II
Gold . . .	Au	197	I III	Silicon . .	Si	28	IV
Helium . .	He	4	—	Silver . .	Ag	108	I
Hydrogen .	H	1	I	Sodium .	Na	23	I
Iodine . .	I	127	I	Sulphur .	S	32	II IV VI
Iron . . .	Fe	56	II III	Tin . . .	Sn	119	II IV
Lead . . .	Pb	207	II IV	Tungsten .	W	184	VI
Magnesium	Mg	24	II	Zinc . .	Zn	65	II

## COMMON UNITS IN THE METRIC AND ENGLISH SYSTEMS

10 millimeters = 1 centimeter (cm.) (mm.)	1 cc. of water at 4° C. weighs 1 gram
100 centimeters = 1 meter (m.)	2.54 centimeters = 1 inch
1000 cubic centimeters (cc.) = 1 liter (l.)	1 meter = 39.37 inches
10 milligrams = 1 centigram (cg.) (mg.)	1 liter = 1.06 quarts
100 centigrams = 1 gram (g.)	1 gram = 15.4 grains
1000 grams = 1 kilogram (kg.)	28.35 grams = 1 ounce
	1 kilogram = 2.20 pounds
	1 pint of water weighs about 1 pound

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# CURRICULUM LABORATORY

# FOUR-FIGURE LOGARITHMS

N	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9



3 9424 03408 2369

N	0	1	2	3	4	5	6	7	8										
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	I	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7530	7543	7551	I	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	I	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	I	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	I	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	I	1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	I	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	I	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	I	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	I	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	I	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	I	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	I	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	I	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	I	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	I	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	I	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	I	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	I	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	I	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	I	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	I	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	I	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	I	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9025	I	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	I	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	I	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	I	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	I	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	I	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	I	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	I	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	O	I	2	2	3	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	O	I	2	2	3	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	O	I	2	2	3	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	O	I	2	2	3	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	O	I	2	2	3	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	O	I	2	2	3	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	O	I	2	2	3	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	O	I	2	2	3	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	O	I	2	2	3	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	O	I	2	2	3	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	O	I	2	2	3	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	O	I	2	2	3	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	O	I	2	2	3	3	3	4	4
	0	1	2	3	4	5	6	7	8	9	I	2	3	4	5	6	7	8	9

